

Development of a Solvent Selection Guide for Aldehyde-based Direct Reductive Amination Processes

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Supporting Information

1. General

1.1. Reagents

All reagents and solvents were obtained from commercial suppliers and were used without further purification unless otherwise stated.

1.2 Experimental Details

All reactions were carried out using conventional glassware at room temperature (generally approx. 18 °C) under an air atmosphere and with no special attention given to the exclusion of moisture.

1.3 Purification of Products

i) Thin layer chromatography was carried out using Merck silica plates coated with fluorescent indicator UV254. These were analysed under 254 nm UV light or developed using potassium permanganate solution.

ii) Flash chromatography was carried out using ZEOPrep 60 HYD 40-63µm silica gel or IST Isolute Flash silica cartridges.

1.4 Analysis of Products

i) Fourier Transformed Infra-Red (FTIR) spectra were obtained on a Shimadzu IRAffinity-1 machine.

ii) ¹H and ¹³C NMR spectra were obtained on a Bruker AV 400 at 400 MHz and 100 MHz respectively, or a Bruker DRX 500 at 500 MHz and 125 MHz, respectively. Chemical shifts are reported in ppm and coupling constants are reported in Hz with CDCl₃ referenced at 7.27 (¹H) and 77.36 ppm (¹³C), respectively.

iii) High-resolution mass spectra were obtained through analysis at the EPSRC National Mass Spectrometry Facility, University of Swansea.

iv) HPLC analysis was carried out on an Agilent Technologies 1200 Series Analytical HPLC using a Macherey-Nagel C₁₈ 5 µM 4.6 x 50 mm column using MeCN/H₂O as the mobile phase with the following gradient:

Time (min)	% MeCN	% H ₂ O
0	5	95
1	50	50
3	50	50
4	90	10
5	5	95
6	5	95

v) Conversions were obtained using an internal standard (iodobenzene or bromobenzene).

2. Experimental Procedures

2.1 Representative Reactions

Reducing Agents	Solvent Selection
NaBH ₃ CN	tert-Butyl methyl ether (TBME)
NaBH(OAc) ₃	Cyclopentyl methyl ether (CPME)
pic-BH ₃	Dichloromethane (CH ₂ Cl ₂)
	1,2-Dichloroethane (DCE)
	Dimethylcarbonate (DMC)
	N,N-Dimethylformamide (DMF)
	Ethyl acetate (EtOAc)
	iso-Propyl alcohol (IPA)
	2-Methyl tetrahydrofuran (2-MeTHF)
	Tetrahydrofuran (THF)

Representative Reactions

Reaction 1: Aryl Aldehyde - Acyclic Primary Aryl Amine

Phenyl aldehyde reacts with phenyl amine in the presence of a reducing agent (1.2 equiv) and solvent at room temperature to form N-phenylphenylamine (1).

Reaction 2: Alkyl Aldehyde - Acyclic Primary Aryl Amine

4-phenylbutanal reacts with phenyl amine in the presence of a reducing agent (1.2 equiv) and solvent at room temperature to form N-(4-phenylbutyl)phenylamine (2).

Reaction 3: Aryl Aldehyde - Acyclic Primary Alkyl Amine

Phenyl aldehyde reacts with propyl amine in the presence of a reducing agent (1.2 equiv) and solvent at room temperature to form N-phenylpropylamine (3).

Reaction 4: Alkyl Aldehyde - Acyclic Primary Alkyl Amine

4-phenylbutanal reacts with propyl amine in the presence of a reducing agent (1.2 equiv) and solvent at room temperature to form N-(4-phenylbutyl)propylamine (4).

Reaction 5: Aryl Aldehyde - Acyclic Secondary Aryl Amine

Phenyl aldehyde reacts with N-methylbenzyl amine in the presence of a reducing agent (1.2 equiv) and solvent at room temperature to form N-phenyl-N-methylbenzylamine (5).

Reaction 6: Alkyl Aldehyde - Acyclic Secondary Aryl Amine

4-phenylbutanal reacts with N-methylbenzyl amine in the presence of a reducing agent (1.2 equiv) and solvent at room temperature to form N-(4-phenylbutyl)-N-methylbenzylamine (6).

Reaction 7: Aryl Aldehyde - Acyclic Secondary Alkyl Amine

Phenyl aldehyde reacts with 4-methoxybenzyl amine in the presence of a reducing agent (1.2 equiv) and solvent at room temperature to form N-phenyl-4-methoxybenzylamine (7).

Reaction 8: Alkyl Aldehyde - Acyclic Secondary Alkyl Amine

4-phenylbutanal reacts with 4-methoxybenzyl amine in the presence of a reducing agent (1.2 equiv) and solvent at room temperature to form N-(4-phenylbutyl)-4-methoxybenzylamine (8).

Reaction 9: Aryl Aldehyde - Cyclic Secondary Aryl Amine

Phenyl aldehyde reacts with 1,2-dihydro-4H-chinolin-4-amine in the presence of a reducing agent (1.2 equiv) and solvent at room temperature to form N-phenyl-1,2-dihydro-4H-chinolin-4-amine (9).

Reaction 10: Alkyl Aldehyde - Cyclic Secondary Aryl Amine

4-phenylbutanal reacts with 1,2-dihydro-4H-chinolin-4-amine in the presence of a reducing agent (1.2 equiv) and solvent at room temperature to form N-(4-phenylbutyl)-1,2-dihydro-4H-chinolin-4-amine (10).

Reaction 11: Aryl Aldehyde - Cyclic Secondary Alkyl Amine

Phenyl aldehyde reacts with 1-phenylpiperazine in the presence of a reducing agent (1.2 equiv) and solvent at room temperature to form N-phenyl-1-phenylpiperazine (11).

Reaction 12: Alkyl Aldehyde - Cyclic Secondary Aryl Amine

4-phenylbutanal reacts with 1-phenylpiperazine in the presence of a reducing agent (1.2 equiv) and solvent at room temperature to form N-(4-phenylbutyl)-1-phenylpiperazine (12).

2.2 General Experimental Procedure for Representative Reactions and Substrate Application

A solution of aldehyde (1 equiv, 0.2 mmol), internal standard, and reducing agent (1.2 equiv, 0.24 mmol) in the appropriate solvent (1 mL, 0.2 M) at room temperature was treated with an amine (1.1 equiv, 0.22 mmol). The reaction was then monitored by HPLC with analyses at 0 h (approx. 5 min), 1 h, 2 h, 4 h, 6 h, 8 h, and 24 h by removal of an aliquot (10 μ L) which was diluted to 1 mL in MeOH before injection. In the case of reactions using NaBH₃CN, several drops of AcOH were added prior to addition of the amine.

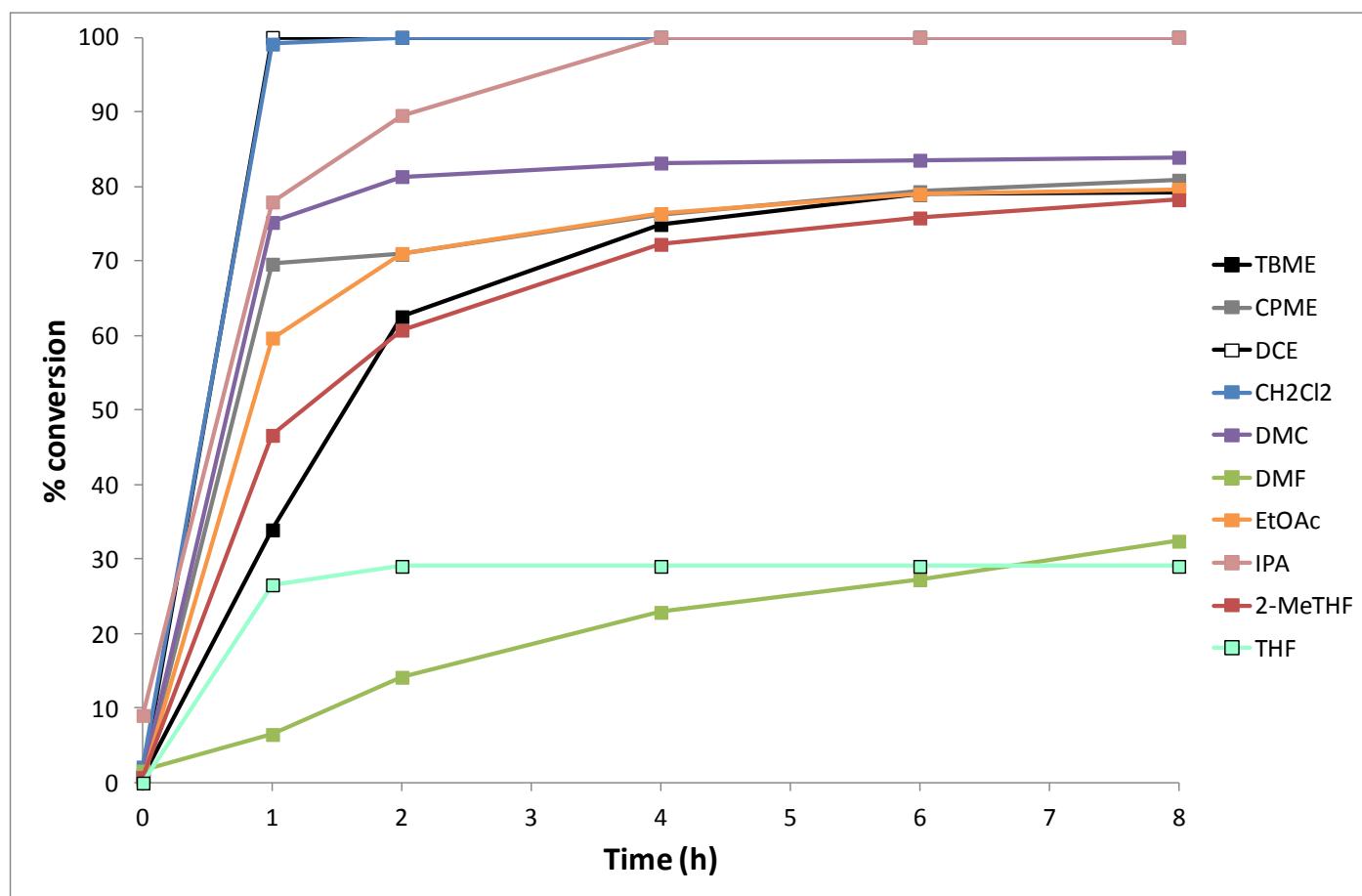
2.3 General Experimental Procedure for Gram-scale Reactions

To a solution of the aldehyde (1 equiv, 20 mmol) in EtOAc (40 mL) was added the amine (1.1 equiv, 22 mmol) followed by NaBH(OAc)₃ (1.2 equiv, 24 mmol) and the mixture was stirred at room temperature for 6 h. The reaction mixture was quenched with a saturated aqueous NaHCO₃ solution (20 mL). The organic layer was separated and the aqueous layer was extracted with ethyl acetate (3 \times 20 mL). The combined organic layers were dried (Na₂SO₄) and concentrated under reduced pressure to a residue, which was purified by flash column chromatography to afford the product.

3. Experimental Data

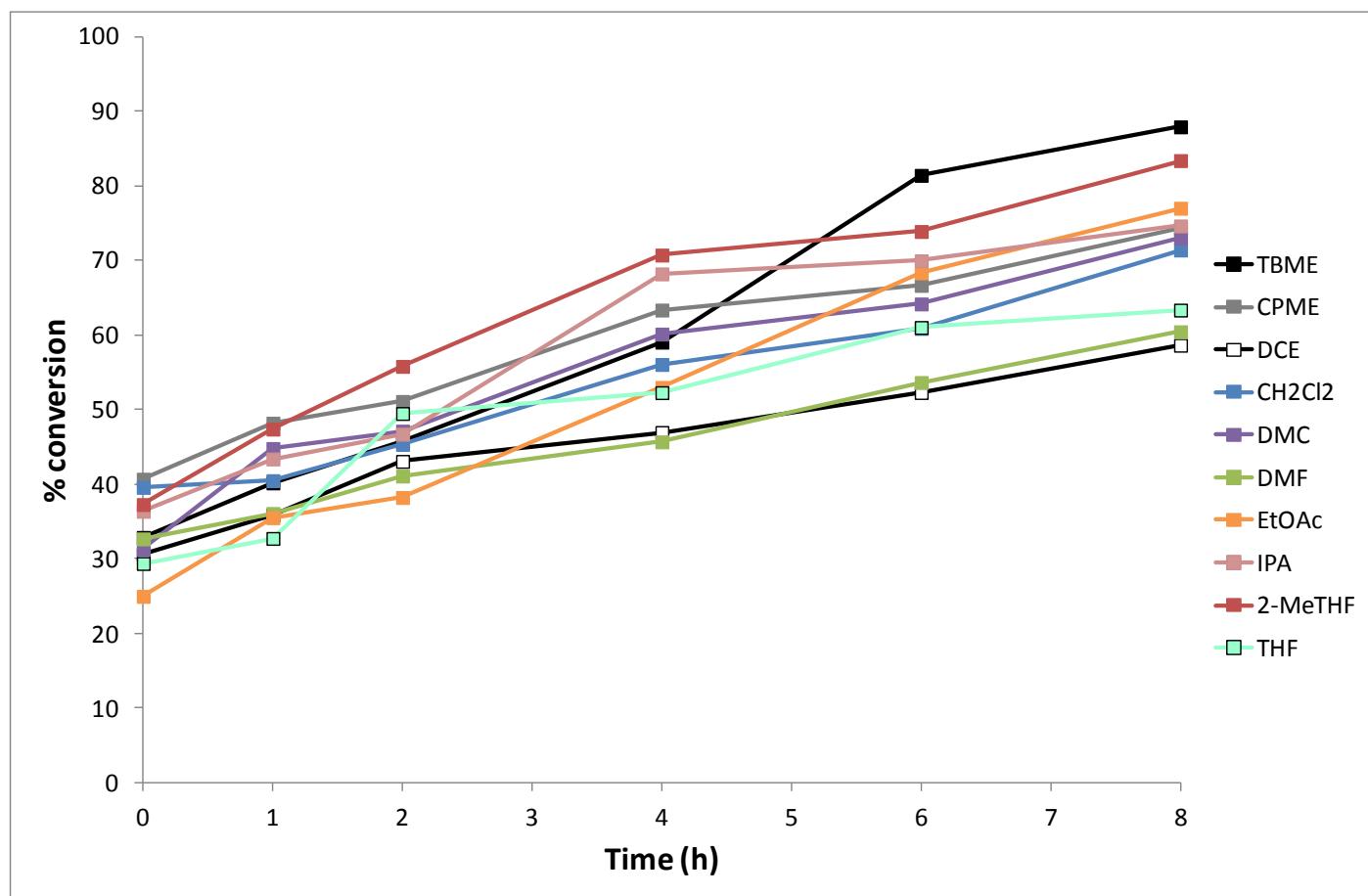
3.1 Conversion vs. Time Data for Reactions 1-12 Using Specific Reducing Agents in the Range of Solvents

Reaction 1: Sodium Triacetoxyborohydride



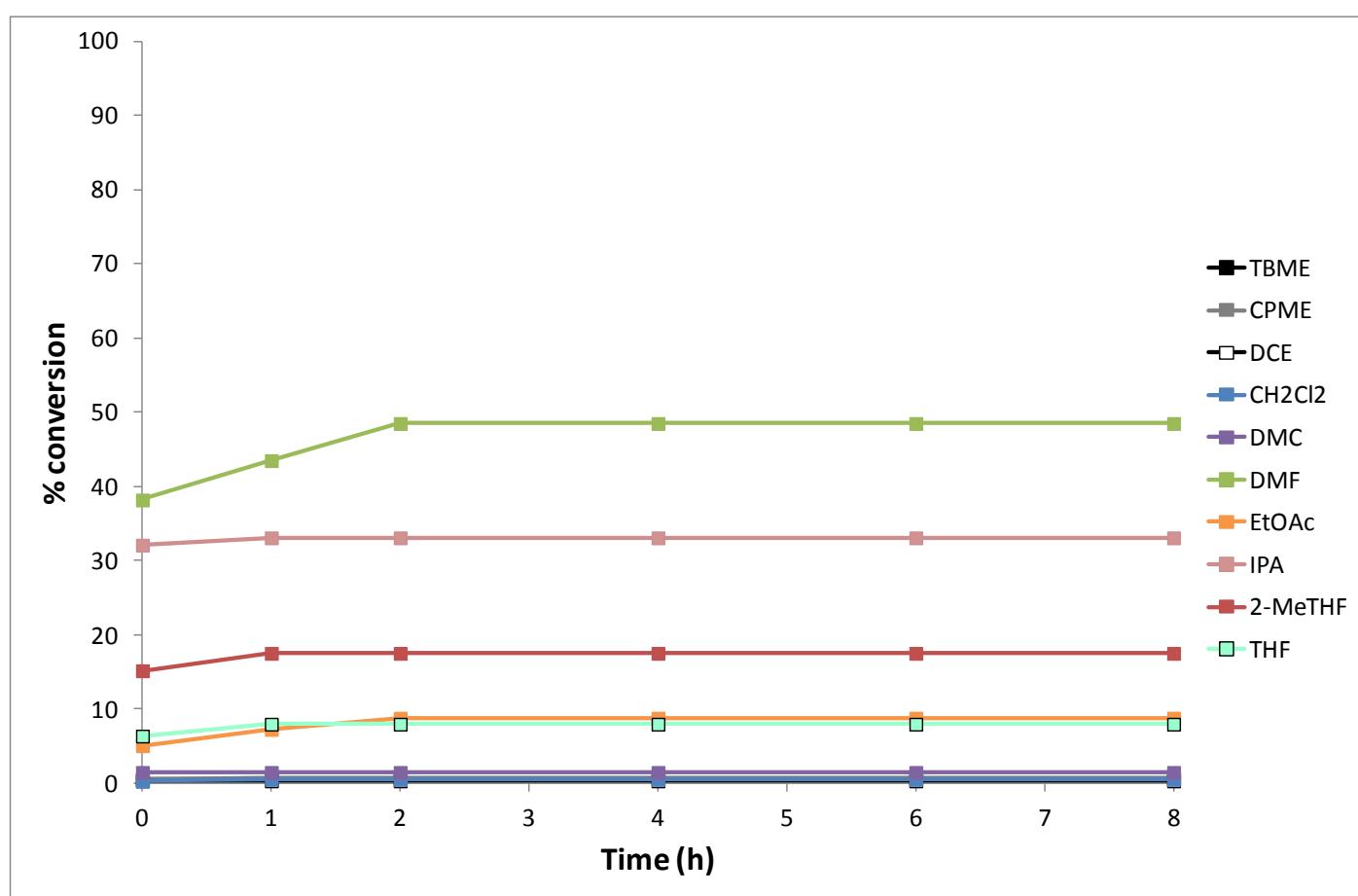
Time (h) / Solvent	TBME	CPME	DCE	CH ₂ Cl ₂	DMC	DMF	EtOAc	IPA	2-MeTHF	THF
0	0.8	0.5	0.9	2.1	0.9	1.6	0.4	9.1	0.7	0.0
1	33.9	69.6	100.0	99.1	75.2	6.5	59.7	77.9	46.6	26.6
2	62.5	70.9	100.0	100.0	81.3	14.2	71.0	89.5	60.7	29.1
4	74.9	76.3	100.0	100.0	83.1	22.9	76.4	100.0	72.2	29.1
6	78.9	79.3	100.0	100.0	83.5	27.3	79.0	100.0	75.8	29.1
8	79.3	80.8	100.0	100.0	83.9	32.4	79.6	100.0	78.2	29.1
24	84.5	85.6	100.0	100.0	85.3	39.3	79.8	100.0	79.6	29.1

Reaction 1: Picoline-borane Complex



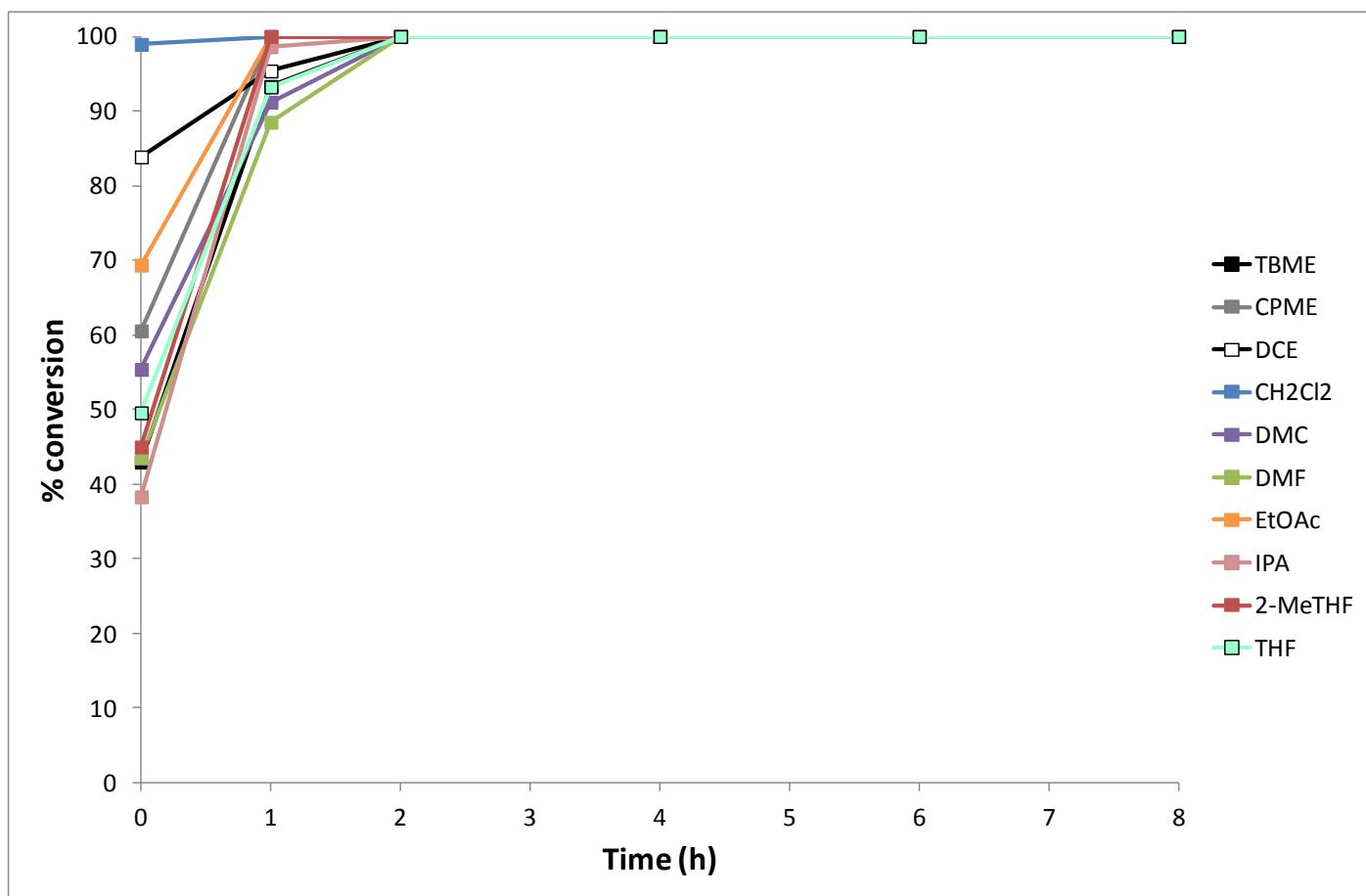
Time (h) / Solvent	TBME	CPME	DCE	CH ₂ Cl ₂	DMC	DMF	EtOAc	IPA	2-MeTHF	THF
0	32.9	40.7	30.6	39.6	31.3	32.7	25.0	36.4	37.3	29.4
1	40.2	48.2	35.9	40.5	44.8	36.1	35.5	43.4	47.4	32.8
2	45.7	51.1	43.1	45.4	47.1	41.1	38.3	46.7	55.8	49.5
4	59.1	63.4	47.0	56.1	60.1	45.7	53.0	68.2	70.7	52.3
6	81.4	66.7	52.3	60.9	64.2	53.6	68.3	70.0	73.9	61.0
8	87.9	74.2	58.6	71.4	73.0	60.4	77.0	74.6	83.4	63.4
24	89.7	80.2	65.0	71.4	79.2	77.1	83.8	74.6	83.4	63.4

Reaction 1: Sodium Cyanoborohydride

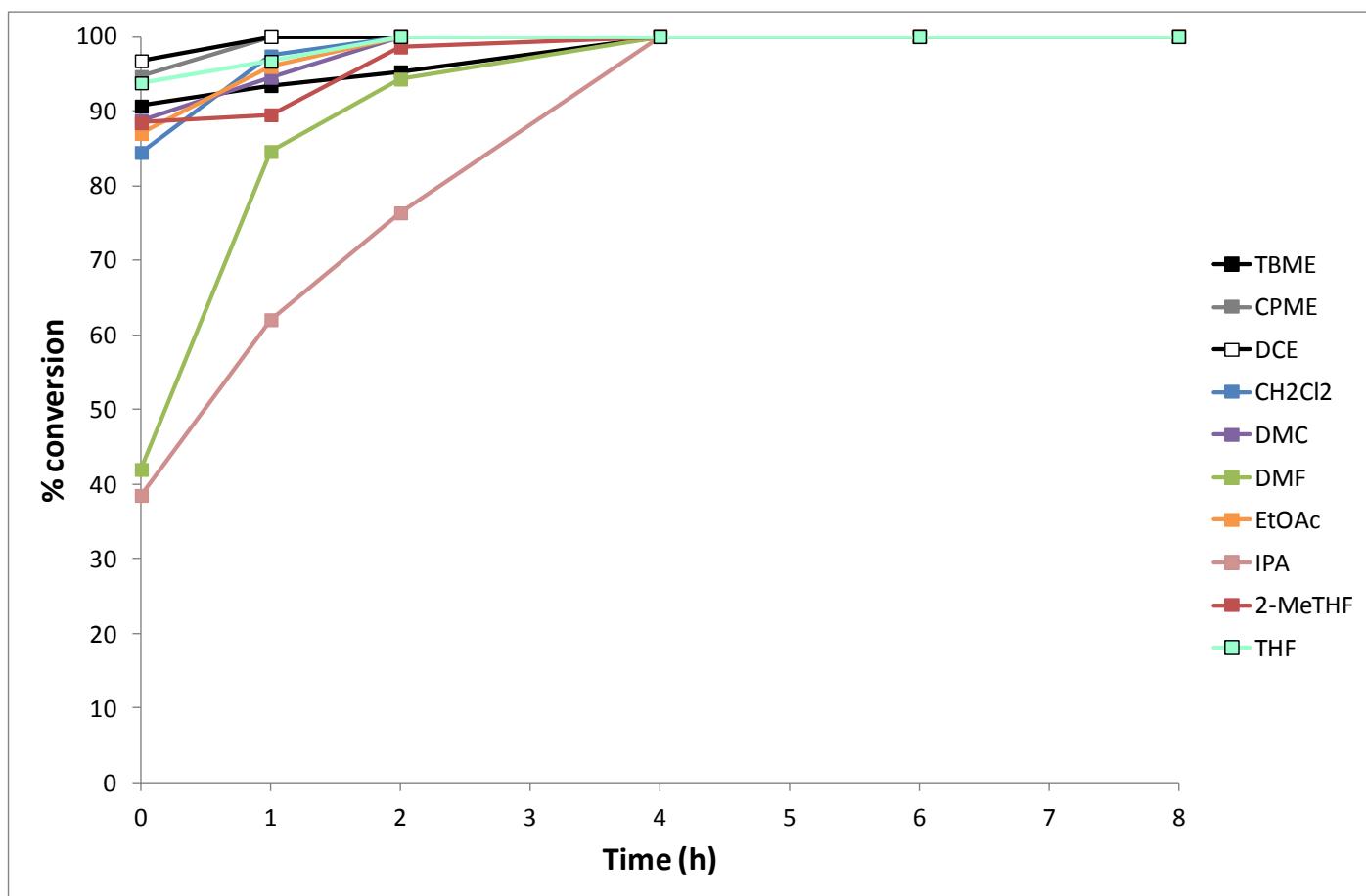


Time (h) / Solvent	TBME	CPME	DCE	CH ₂ Cl ₂	DMC	DMF	EtOAc	IPA	2-MeTHF	THF
0	0.5	0.5	0.4	0.3	1.5	38.2	5.1	32.1	15.2	6.4
1	0.5	0.7	0.4	0.6	1.5	43.5	7.3	33.1	17.5	8.0
2	0.5	0.7	0.4	0.6	1.5	48.5	8.8	33.1	17.5	8.0
4	0.5	0.7	0.4	0.6	1.5	48.5	8.8	33.1	17.5	8.0
6	0.5	0.7	0.4	0.6	1.5	48.5	8.8	33.1	17.5	8.0
8	0.5	0.7	0.4	0.6	1.5	48.5	8.8	33.1	17.5	8.0
24	0.5	0.7	0.4	0.6	1.5	48.5	8.8	33.1	17.5	8.0

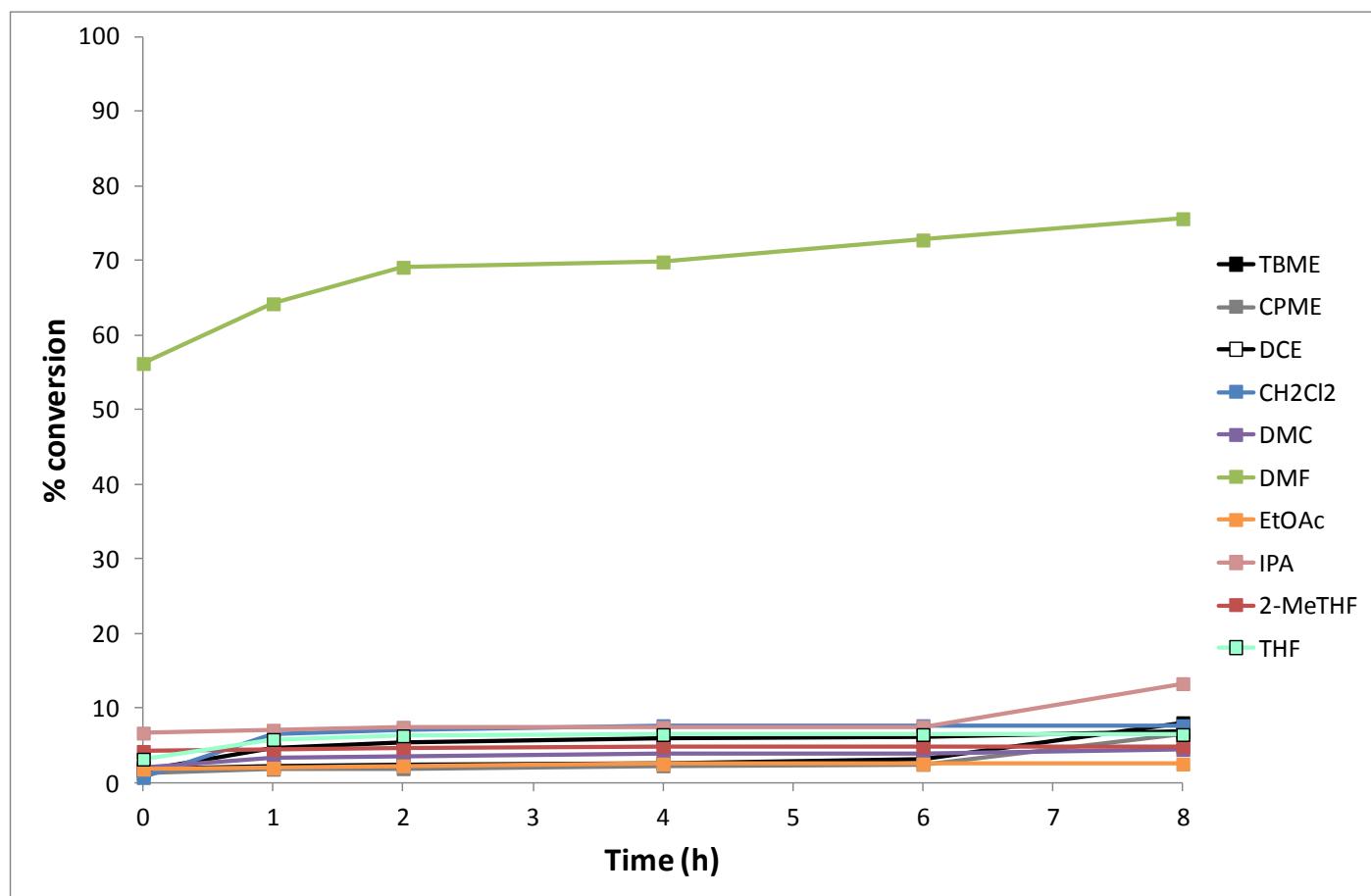
Reaction 2: Sodium Triacetoxyborohydride



Reaction 2: Picoline-borane Complex

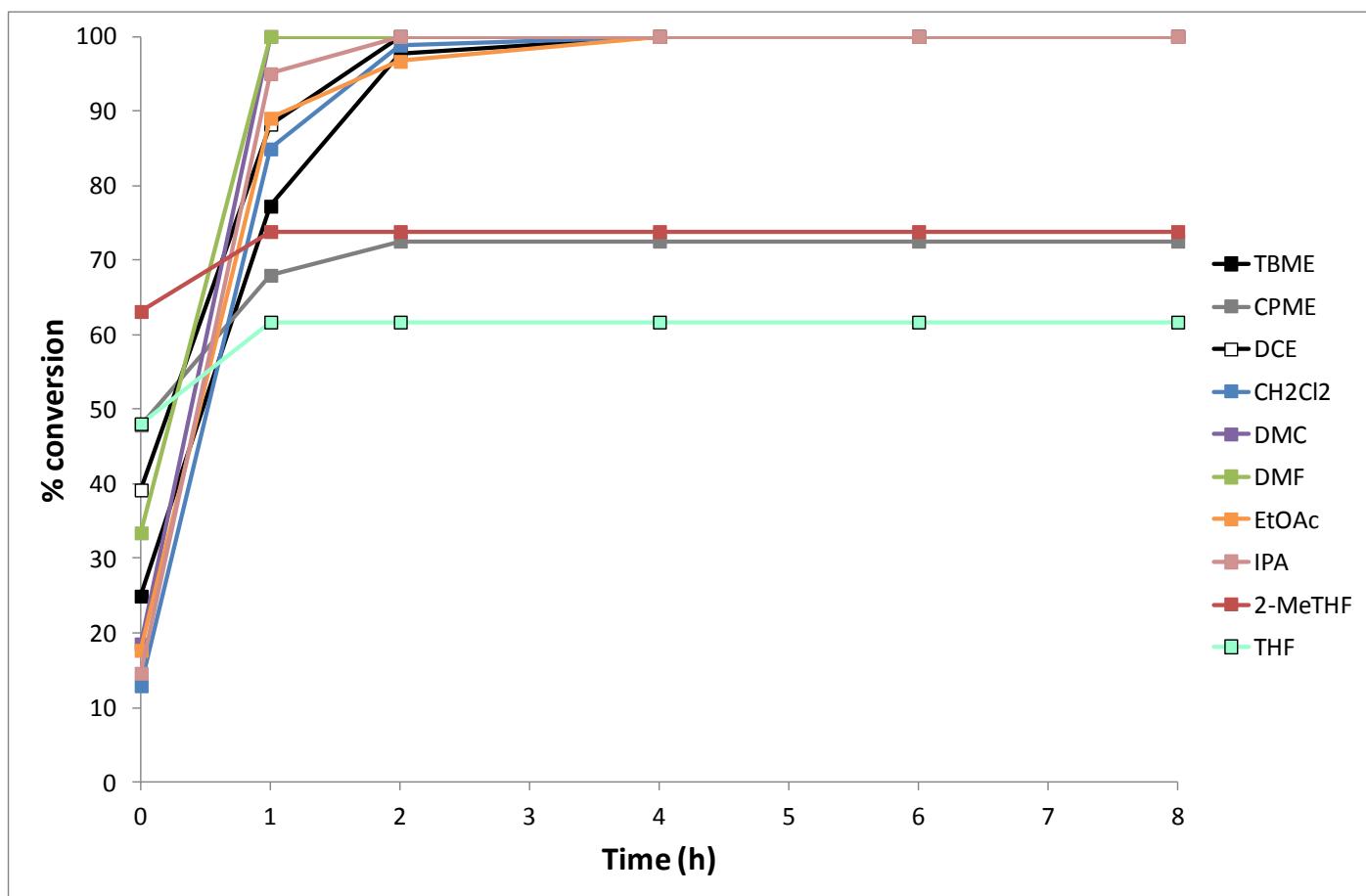


Reaction 2: Sodium Cyanoborohydride



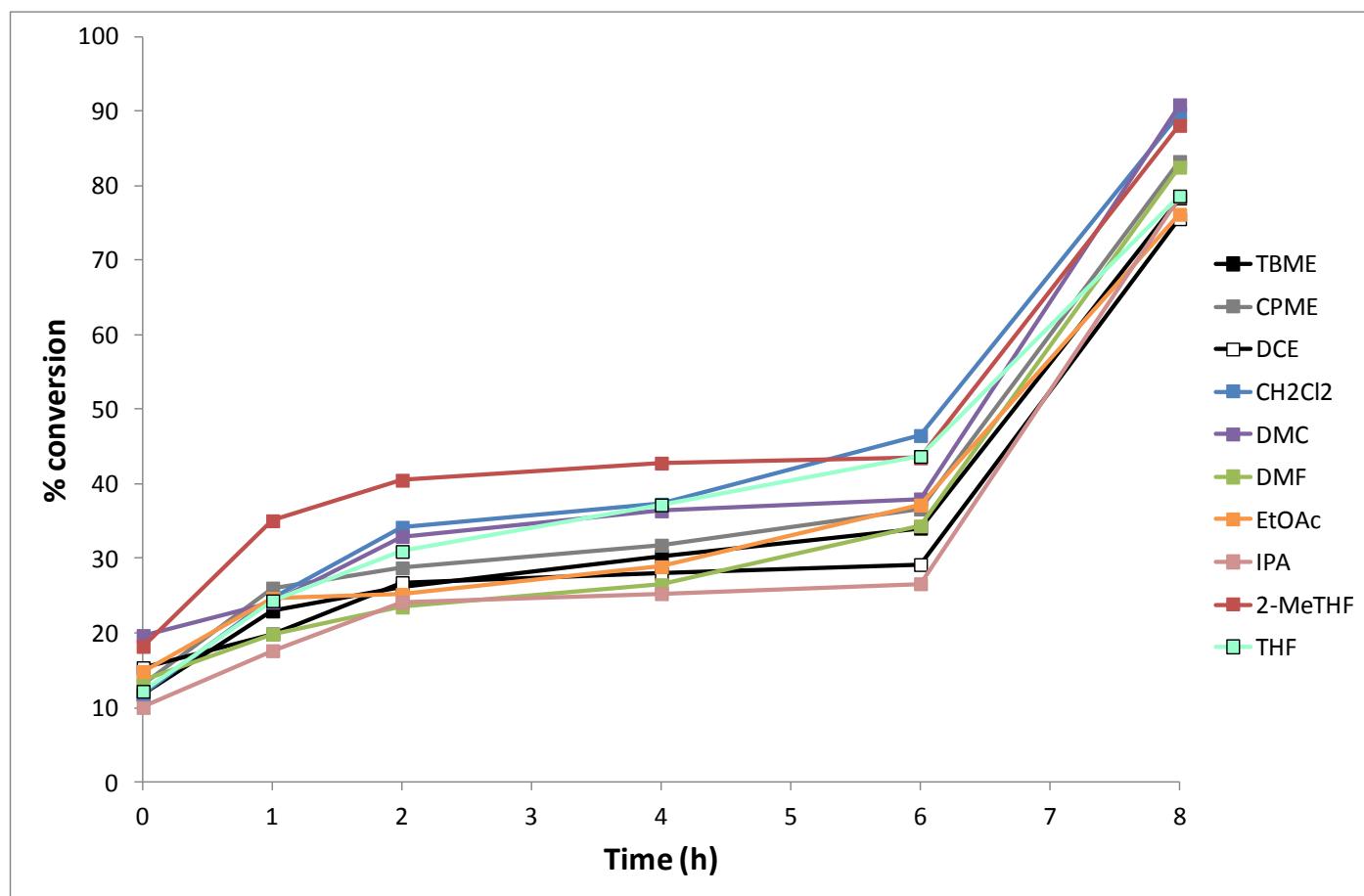
Time (h) / Solvent	TBME	CPME	DCE	CH ₂ Cl ₂	DMC	DMF	EtOAc	IPA	2-MeTHF	THF
0	1.8	1.3	1.7	0.7	2.1	56.2	1.9	6.7	4.2	3.2
1	2.2	1.8	4.6	6.5	3.3	64.2	2.0	7.1	4.5	5.8
2	2.5	1.9	5.3	7.1	3.6	69.1	2.3	7.5	4.7	6.3
4	2.7	2.3	5.9	7.7	3.9	69.8	2.5	7.5	4.9	6.5
6	3.1	2.5	6.2	7.7	4.0	72.7	2.5	7.5	4.9	6.5
8	8.1	6.4	6.8	7.7	4.5	75.6	2.5	13.3	4.9	6.5
24	12.6	8.5	6.8	7.7	5.5	100.0	2.5	13.3	4.9	6.5

Reaction 3: Sodium Triacetoxyborohydride



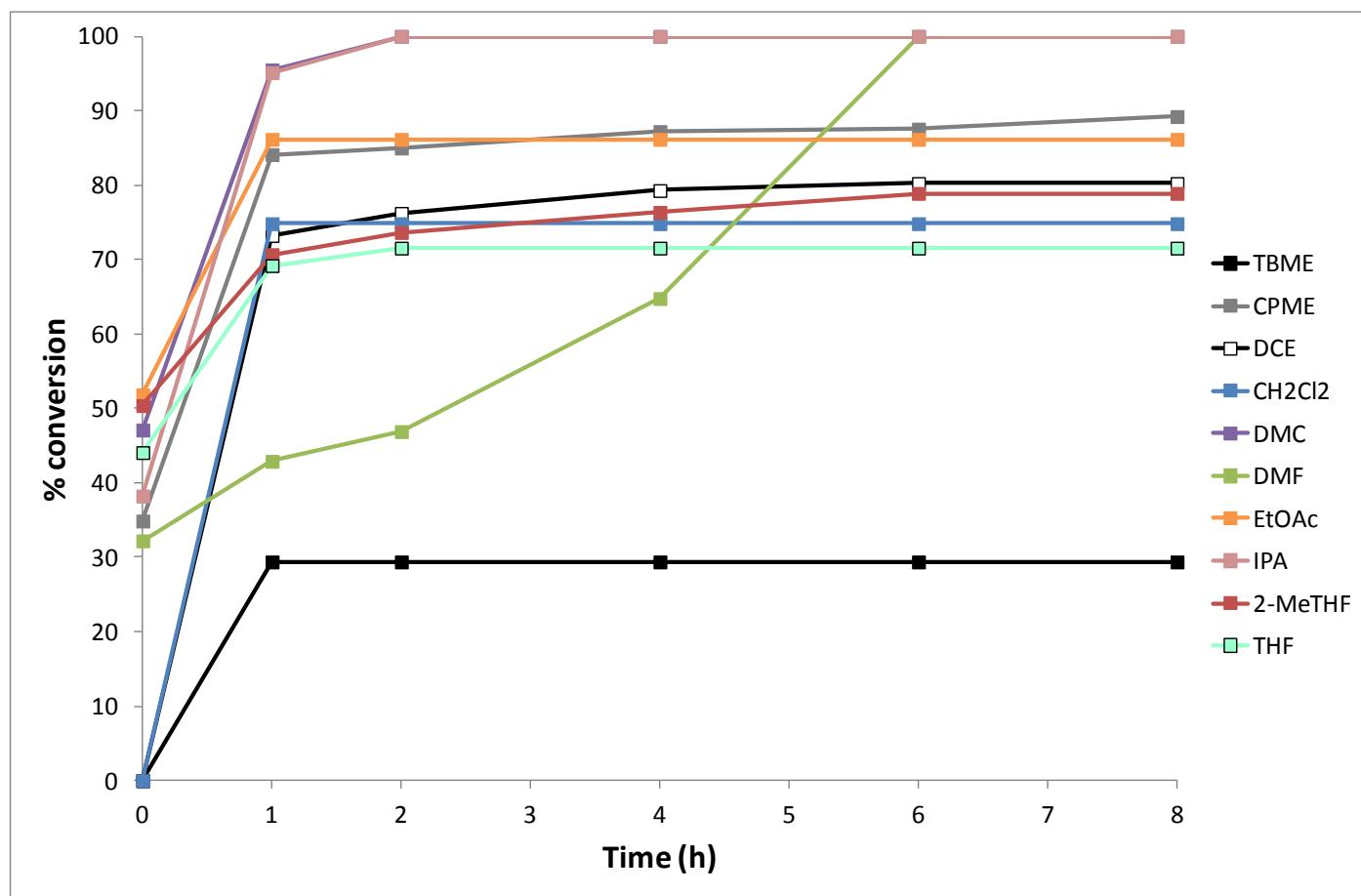
Time (h) / Solvent	TBME	CPME	DCE	CH ₂ Cl ₂	DMC	DMF	EtOAc	IPA	2-MeTHF	THF
0	25.0	47.9	39.2	12.9	18.6	33.4	17.7	14.6	63.1	48.1
1	77.3	68.0	88.2	84.9	100.0	100.0	89.0	95.0	73.9	61.7
2	97.8	72.6	100.0	98.8	100.0	100.0	96.7	100.0	73.9	61.7
4	100.0	72.6	100.0	100.0	100.0	100.0	100.0	100.0	73.9	61.7
6	100.0	72.6	100.0	100.0	100.0	100.0	100.0	100.0	73.9	61.7
8	100.0	72.6	100.0	100.0	100.0	100.0	100.0	100.0	73.9	61.7
24	100.0	72.6	100.0	100.0	100.0	100.0	100.0	100.0	73.9	61.7

Reaction 3: Picoline-borane Complex



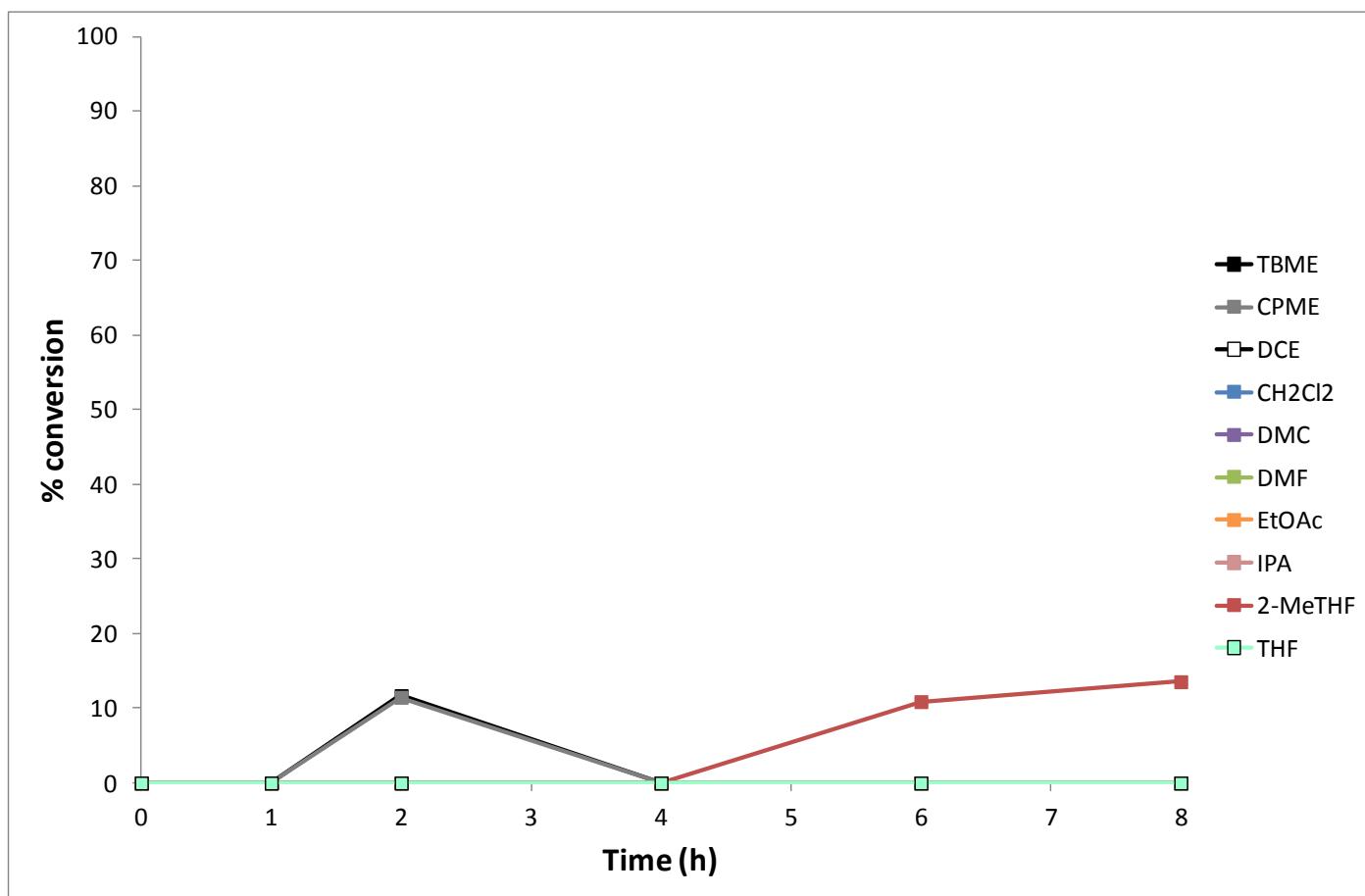
Time (h) / Solvent	TBME	CPME	DCE	CH ₂ Cl ₂	DMC	DMF	EtOAc	IPA	2-MeTHF	THF
0	11.8	13.1	15.4	11.8	19.7	13.6	14.9	10.0	18.2	12.2
1	23.0	26.1	19.9	24.6	24.2	19.9	24.7	17.7	35.1	24.3
2	26.2	28.7	26.8	34.2	33.0	23.5	25.2	24.2	40.5	31.0
4	30.3	31.8	28.0	37.3	36.4	26.5	28.9	25.3	42.8	37.2
6	34.1	36.6	29.2	46.5	38.0	34.4	37.2	26.6	43.5	43.7
8	78.3	83.2	75.6	89.7	90.8	82.5	76.2	78.5	88.1	78.6
24	78.3	83.2	75.6	89.7	93.2	53.5	76.2	78.5	88.1	78.6

Reaction 3: Sodium Cyanoborohydride



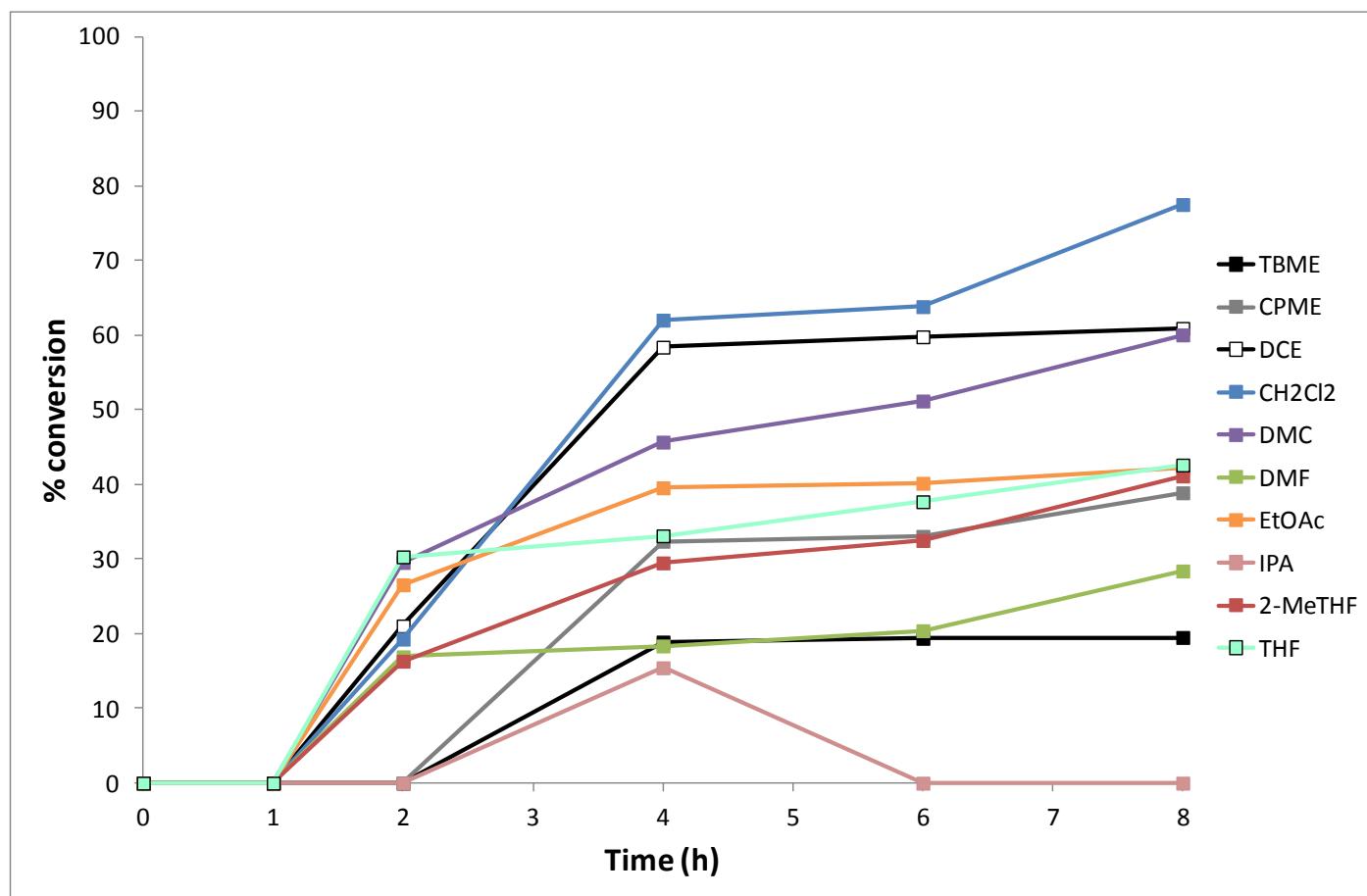
Time (h) / Solvent	TBME	CPME	DCE	CH ₂ Cl ₂	DMC	DMF	EtOAc	IPA	2-MeTHF	THF
0	0.0	34.9	0.0	0.0	47.1	32.2	51.8	38.2	50.4	44.1
1	29.4	84.1	73.2	74.8	95.5	42.9	86.2	95.1	70.7	69.2
2	29.4	85.0	76.2	74.8	100.0	46.9	86.2	100.0	73.7	71.6
4	29.4	87.2	79.3	74.8	100.0	64.8	86.2	100.0	76.4	71.6
6	29.4	87.5	80.2	74.8	100.0	100.0	86.2	100.0	78.9	71.6
8	29.4	89.2	80.2	74.8	100.0	100.0	86.2	100.0	78.9	71.6
24	29.4	100.0	80.2	74.8	100.0	100.0	86.2	100.0	78.9	71.6

Reaction 4: Sodium Triacetoxyborohydride



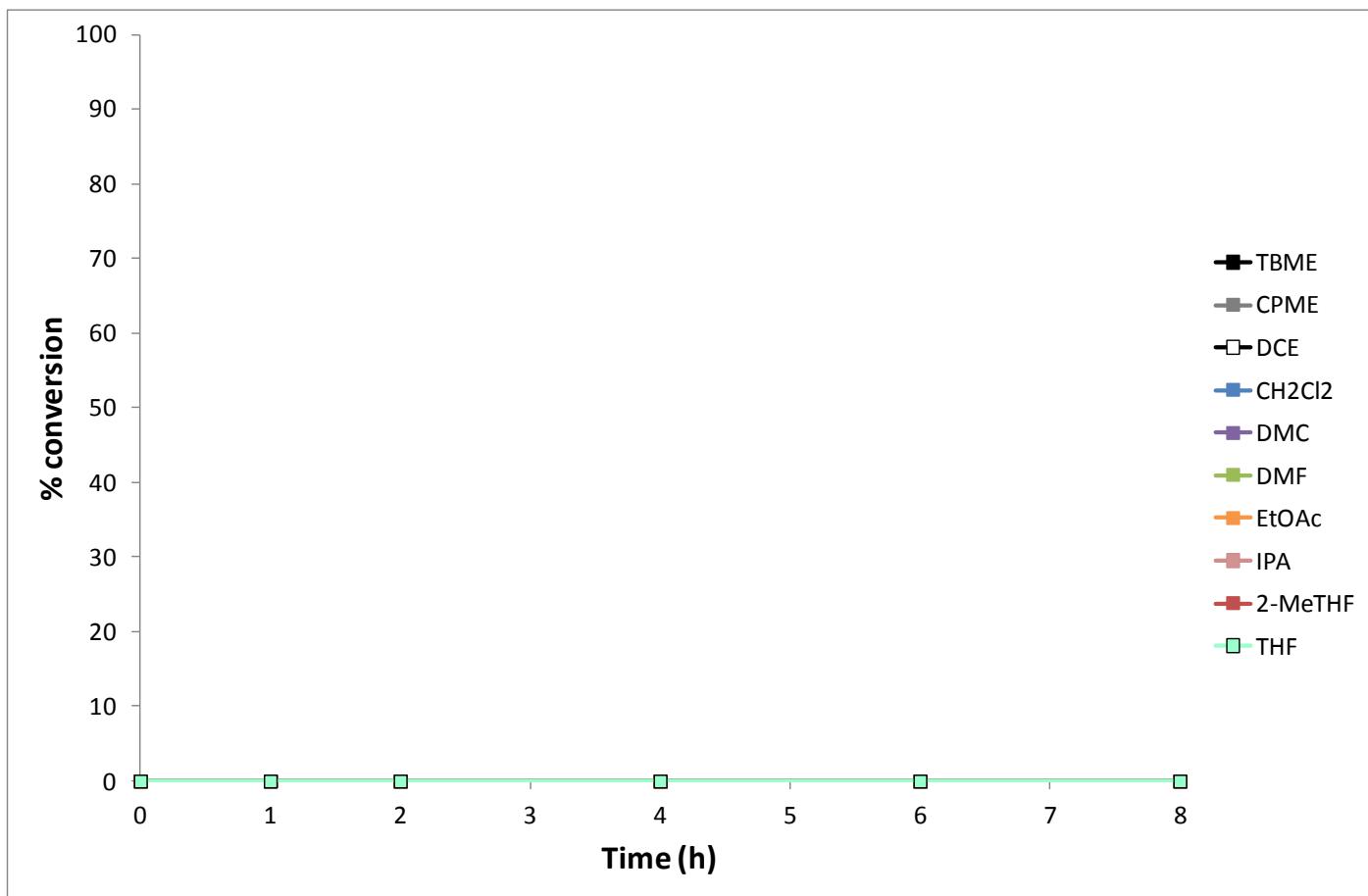
Time (h) / Solvent	TBME	CPME	DCE	CH ₂ Cl ₂	DMC	DMF	EtOAc	IPA	2-MeTHF	THF
0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
1	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
2	11.7	11.5	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
4	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
6	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	10.9	0.0
8	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	13.6	0.0
24	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	13.6	0.0

Reaction 4: Picoline-borane Complex

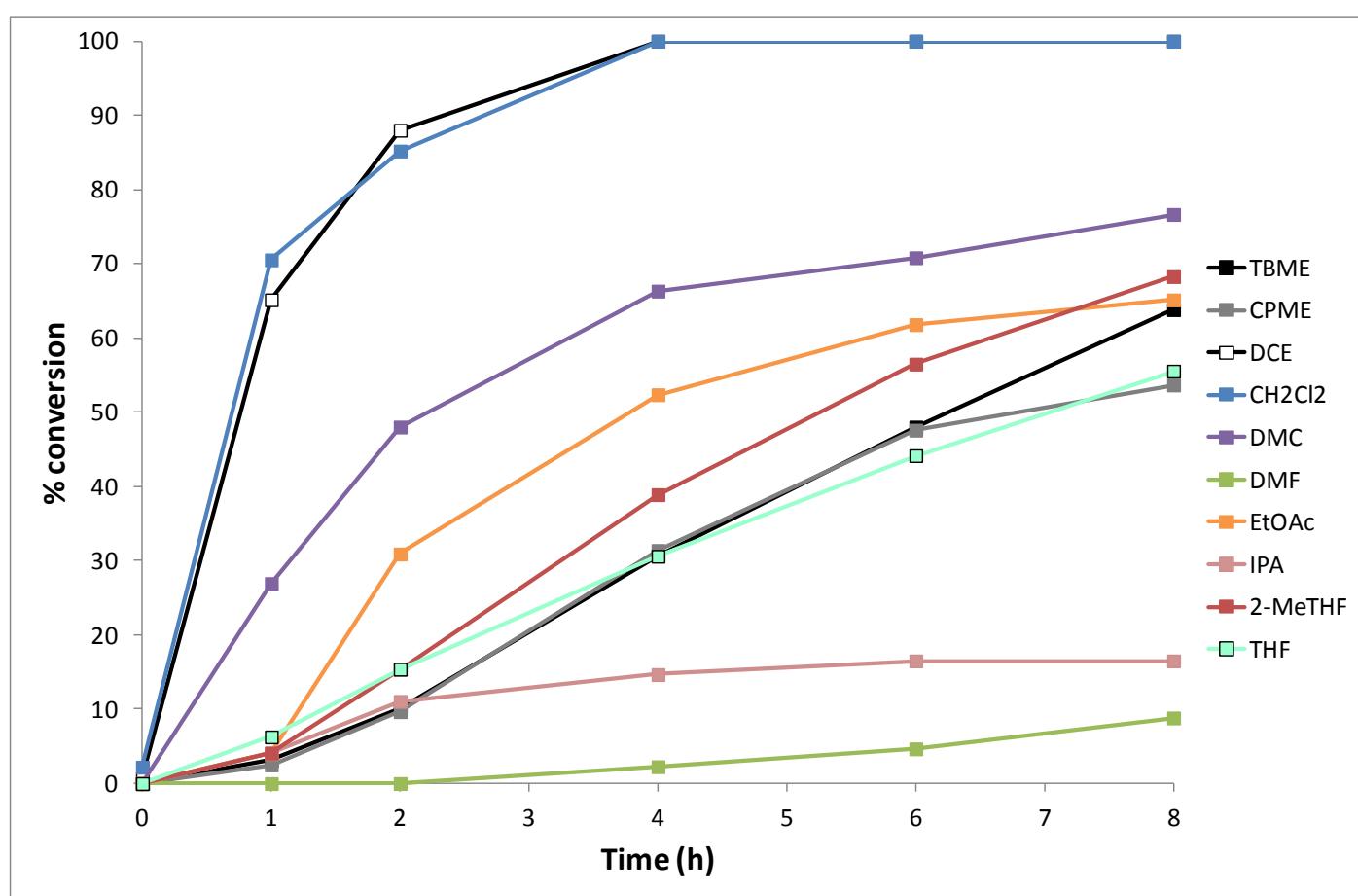


Time (h) / Solvent	TBME	CPME	DCE	CH ₂ Cl ₂	DMC	DMF	EtOAc	IPA	2-MeTHF	THF
0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
1	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
2	0.0	0.0	21.1	19.3	29.6	16.9	26.6	0.0	16.3	30.3
4	18.9	32.4	58.4	62.0	45.7	18.4	39.6	15.5	29.5	33.1
6	19.4	33.1	59.8	63.8	51.2	20.4	40.2	0.0	32.5	37.7
8	19.5	38.9	60.9	77.5	60.0	28.4	42.3	0.0	41.1	42.6
24	19.5	38.9	60.9	77.5	60.0	28.4	42.3	0.0	41.1	42.6

Reaction 4: Sodium Cyanoborohydride

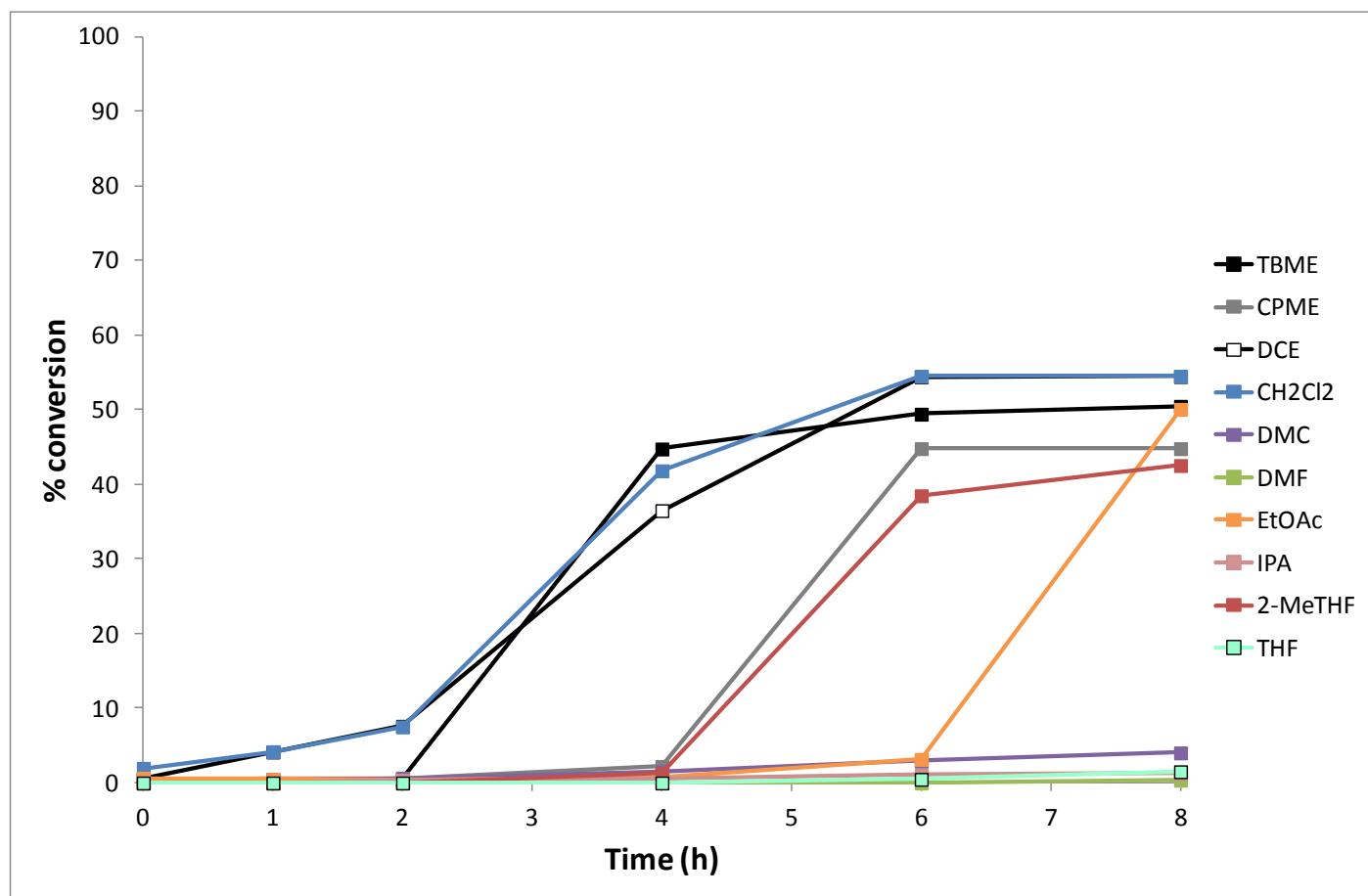


Reaction 5: Sodium Triacetoxyborohydride



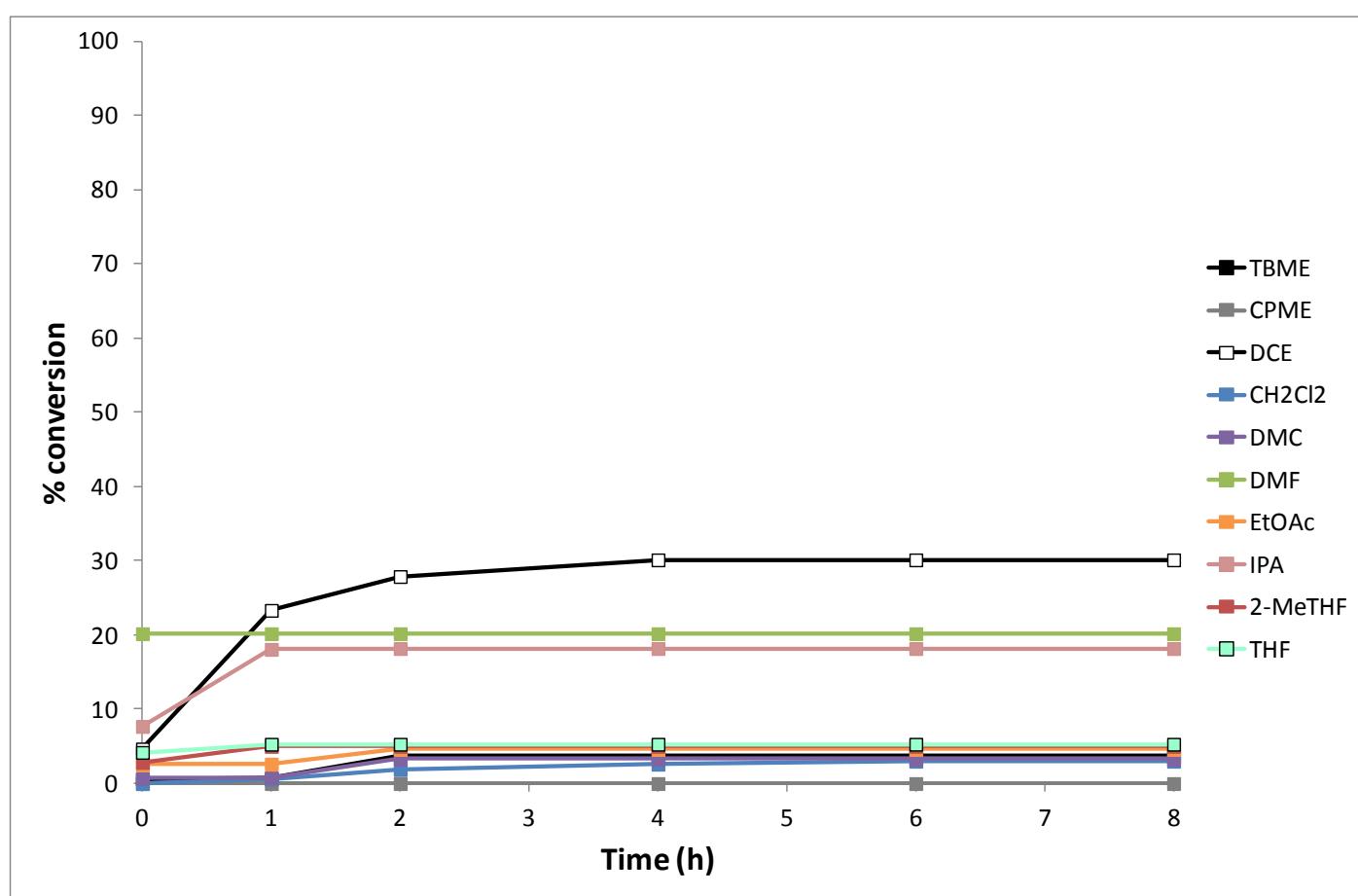
Time (h) / Solvent	TBME	CPME	DCE	CH ₂ Cl ₂	DMC	DMF	EtOAc	IPA	2-MeTHF	THF
0	0.0	0.0	0.7	2.2	0.0	0.0	0.0	0.0	0.0	0.0
1	3.1	2.5	65.2	70.5	26.9	0.0	4.0	4.1	4.1	6.3
2	10.2	9.7	88.0	85.2	48.0	0.0	30.9	11.1	15.4	15.4
4	30.7	31.4	100.0	100.0	66.3	2.3	52.4	14.7	38.9	30.6
6	48.0	47.6	100.0	100.0	70.8	4.7	61.8	16.5	56.5	44.2
8	63.8	53.7	100.0	100.0	76.7	8.8	65.2	16.5	68.3	55.6
24	89.4	87.4	100.0	100.0	87.1	25.4	72.0	17.0	73.1	57.7

Reaction 5: Picoline-borane Complex



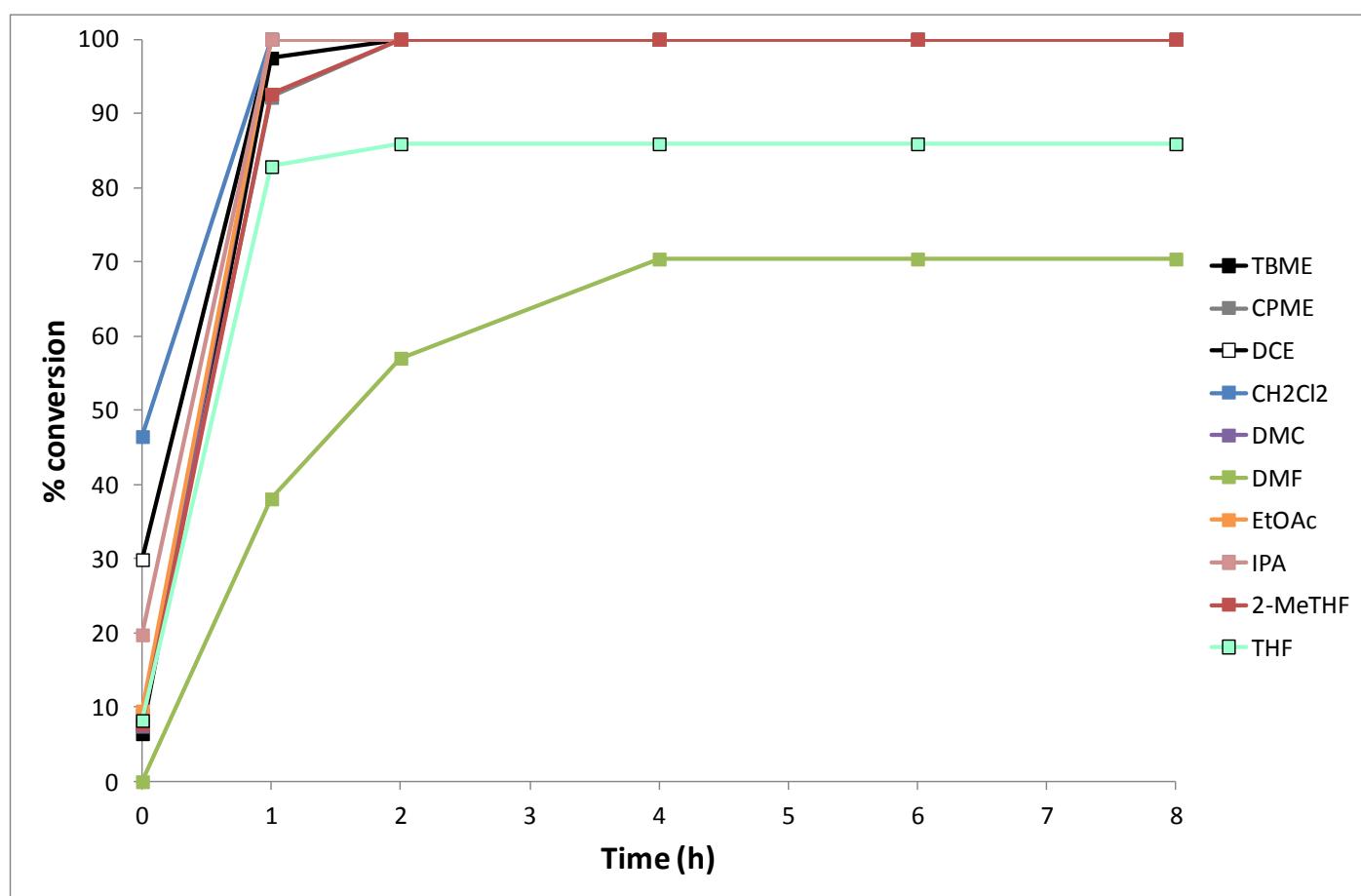
Time (h) / Solvent	TBME	CPME	DCE	CH ₂ Cl ₂	DMC	DMF	EtOAc	IPA	2-MeTHF	THF
0	0.0	0.4	0.6	2.0	0.4	0.0	0.6	0.0	0.0	0.0
1	0.4	0.0	4.1	4.2	0.5	0.0	0.5	0.0	0.0	0.0
2	0.5	0.6	7.6	7.5	0.7	0.0	0.4	0.4	0.0	0.0
4	44.7	2.2	36.5	41.8	1.5	0.0	0.7	0.6	1.2	0.0
6	49.4	44.8	54.4	54.5	2.9	0.0	3.2	1.0	38.5	0.5
8	50.5	44.8	54.4	54.5	4.0	0.4	50.1	1.2	42.5	1.5
24	50.5	44.8	54.4	54.5	47.8	8.6	52.3	6.1	42.5	48.9

Reaction 5: Sodium Cyanoborohydride



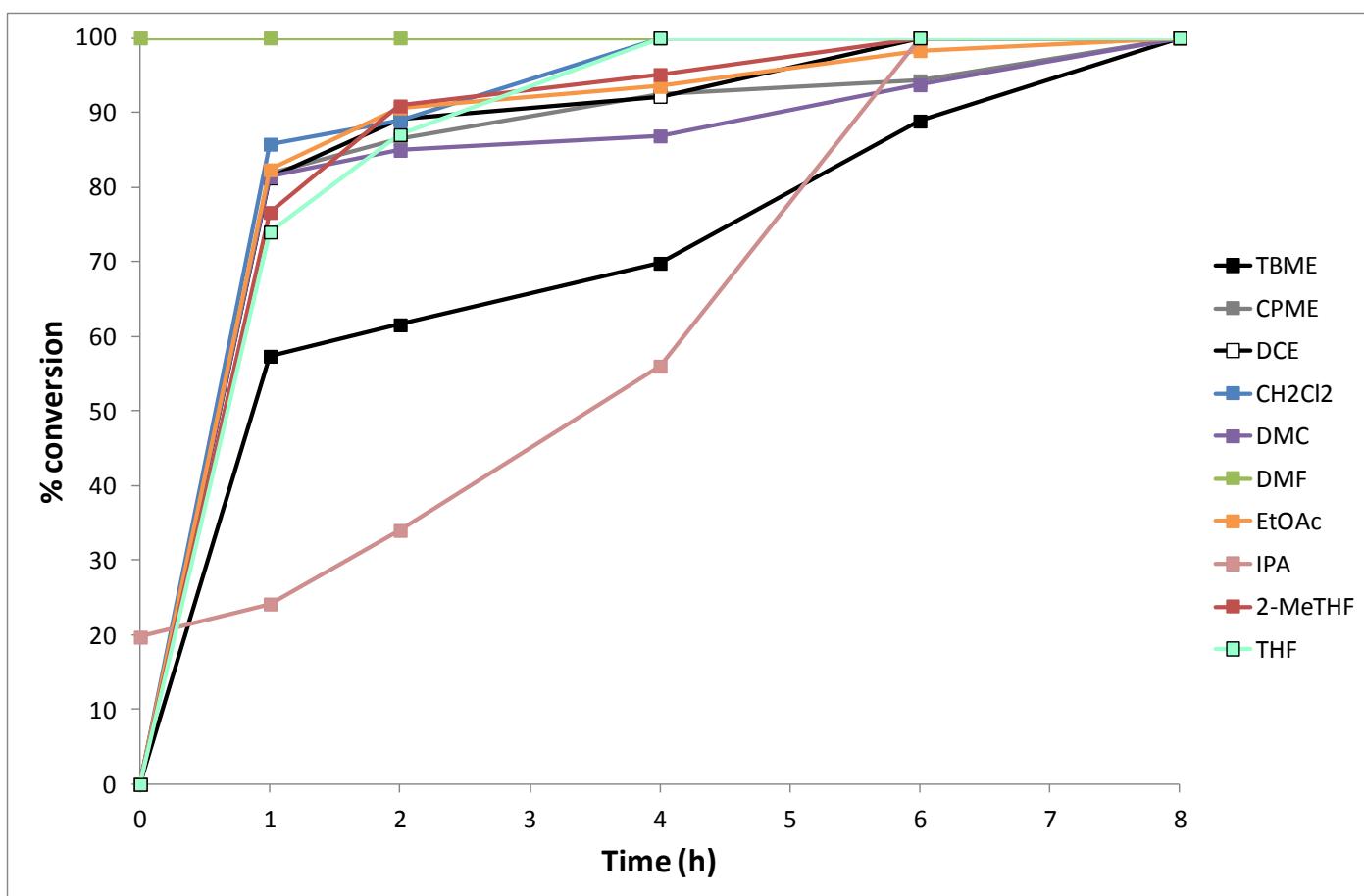
Time (h) / Solvent	TBME	CPME	DCE	CH ₂ Cl ₂	DMC	DMF	EtOAc	IPA	2-MeTHF	THF
0	0.6	0.0	4.7	0.0	0.7	20.2	2.5	7.7	2.8	4.2
1	0.7	0.0	23.3	0.6	0.8	20.2	2.6	18.1	5.0	5.3
2	3.7	0.0	27.9	1.9	3.3	20.2	4.6	18.2	5.0	5.3
4	3.7	0.0	30.1	2.6	3.3	20.2	4.6	18.2	5.0	5.3
6	3.7	0.0	30.1	3.0	3.3	20.2	4.6	18.2	5.0	5.3
8	3.7	0.0	30.1	3.0	3.3	20.2	4.6	18.2	5.0	5.3
24	3.7	0.0	30.1	3.0	3.3	20.2	4.6	18.2	5.0	5.3

Reaction 6: Sodium Triacetoxyborohydride

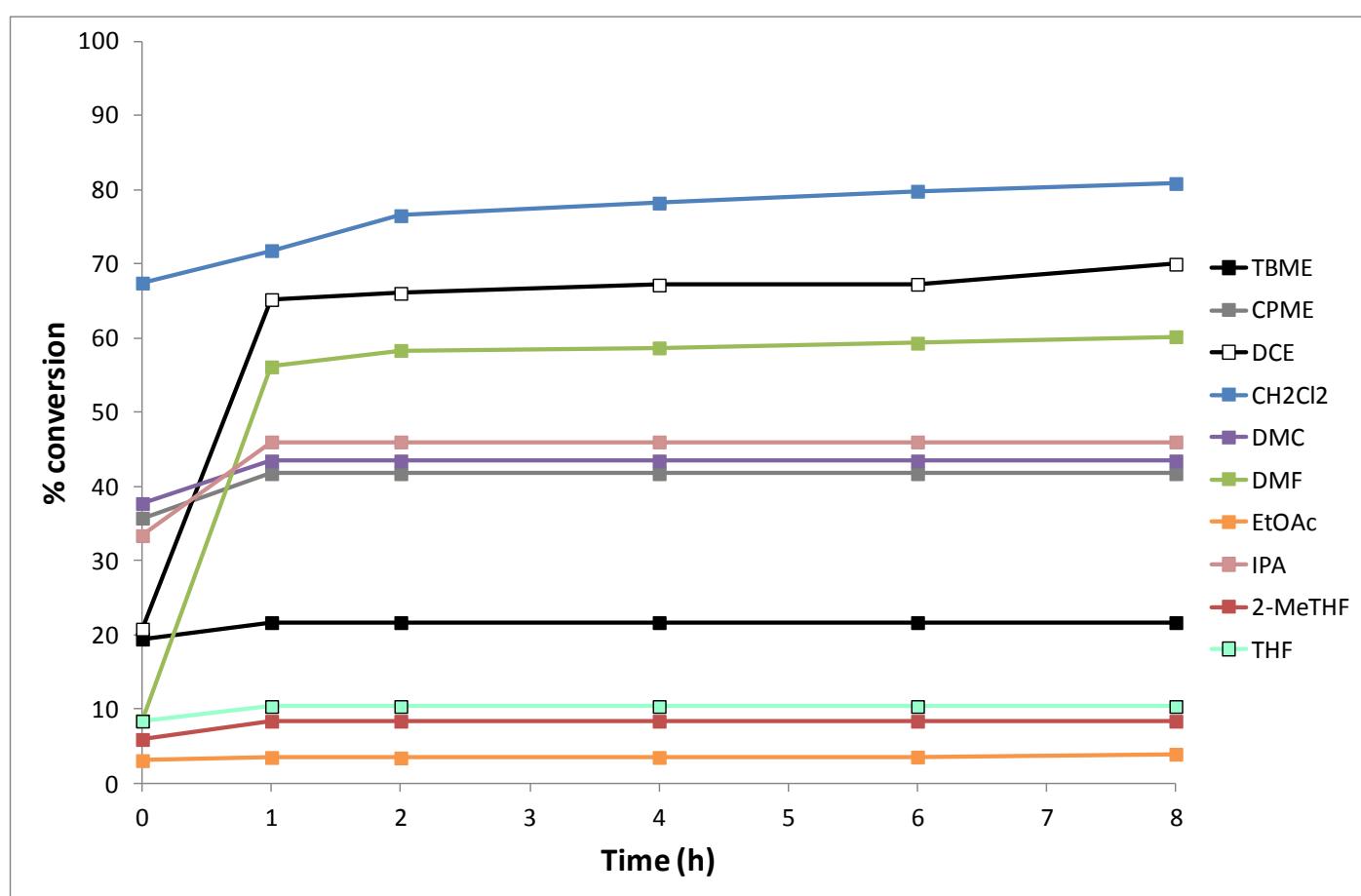


Time (h) / Solvent	TBME	CPME	DCE	CH ₂ Cl ₂	DMC	DMF	EtOAc	IPA	2-MeTHF	THF
0	6.5	9.0	29.9	46.5	7.5	0.0	9.5	19.8	7.7	8.2
1	97.5	92.2	100.0	100.0	100.0	38.1	100.0	100.0	92.6	82.8
2	100.0	100.0	100.0	100.0	100.0	57.0	100.0	100.0	100.0	85.9
4	100.0	100.0	100.0	100.0	100.0	70.4	100.0	100.0	100.0	85.9
6	100.0	100.0	100.0	100.0	100.0	70.4	100.0	100.0	100.0	85.9
8	100.0	100.0	100.0	100.0	100.0	70.4	100.0	100.0	100.0	85.9
24	100.0	100.0	100.0	100.0	100.0	70.4	100.0	100.0	100.0	85.9

Reaction 6: Picoline-borane Complex

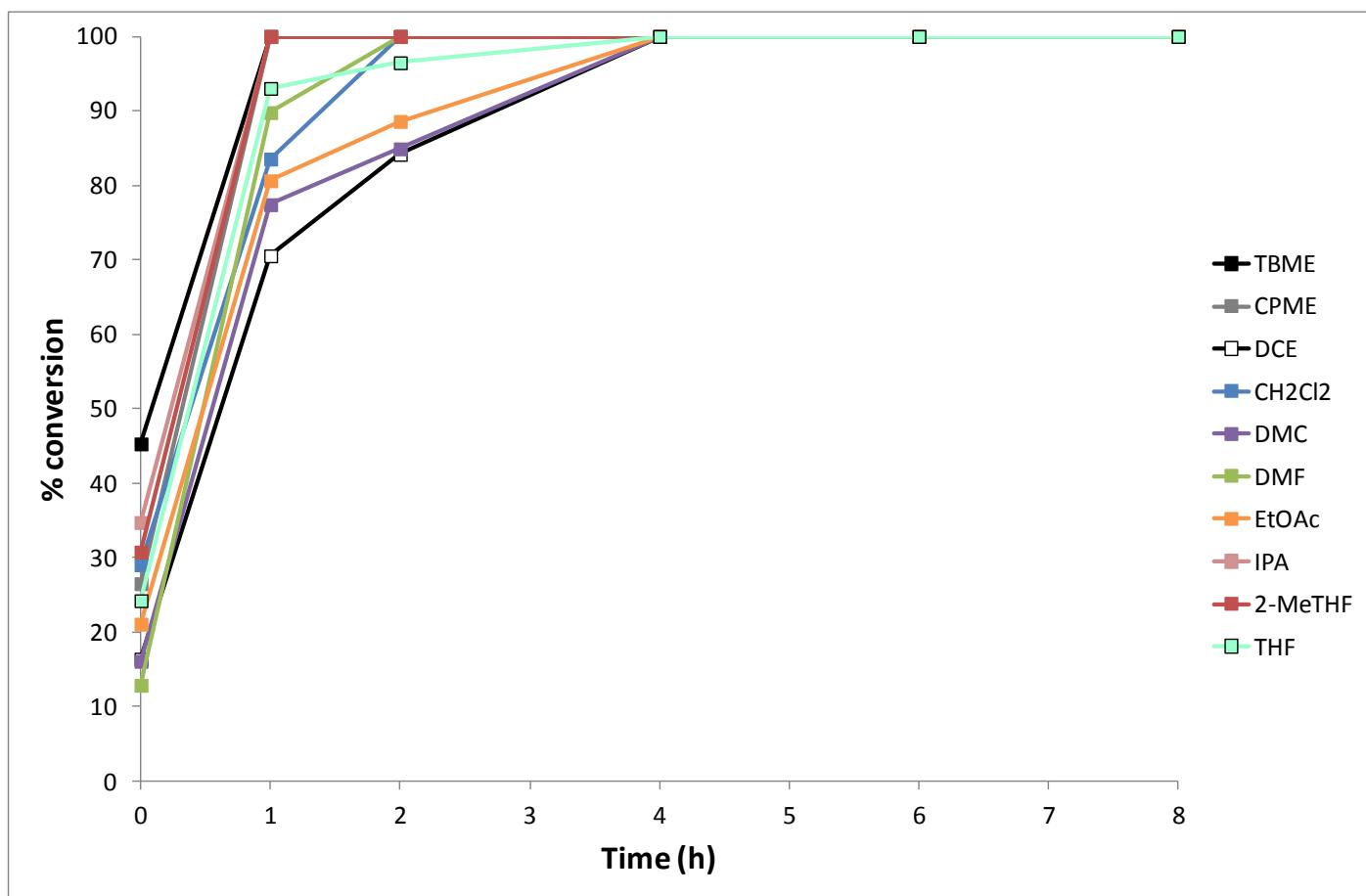


Reaction 6: Sodium Cyanoborohydride

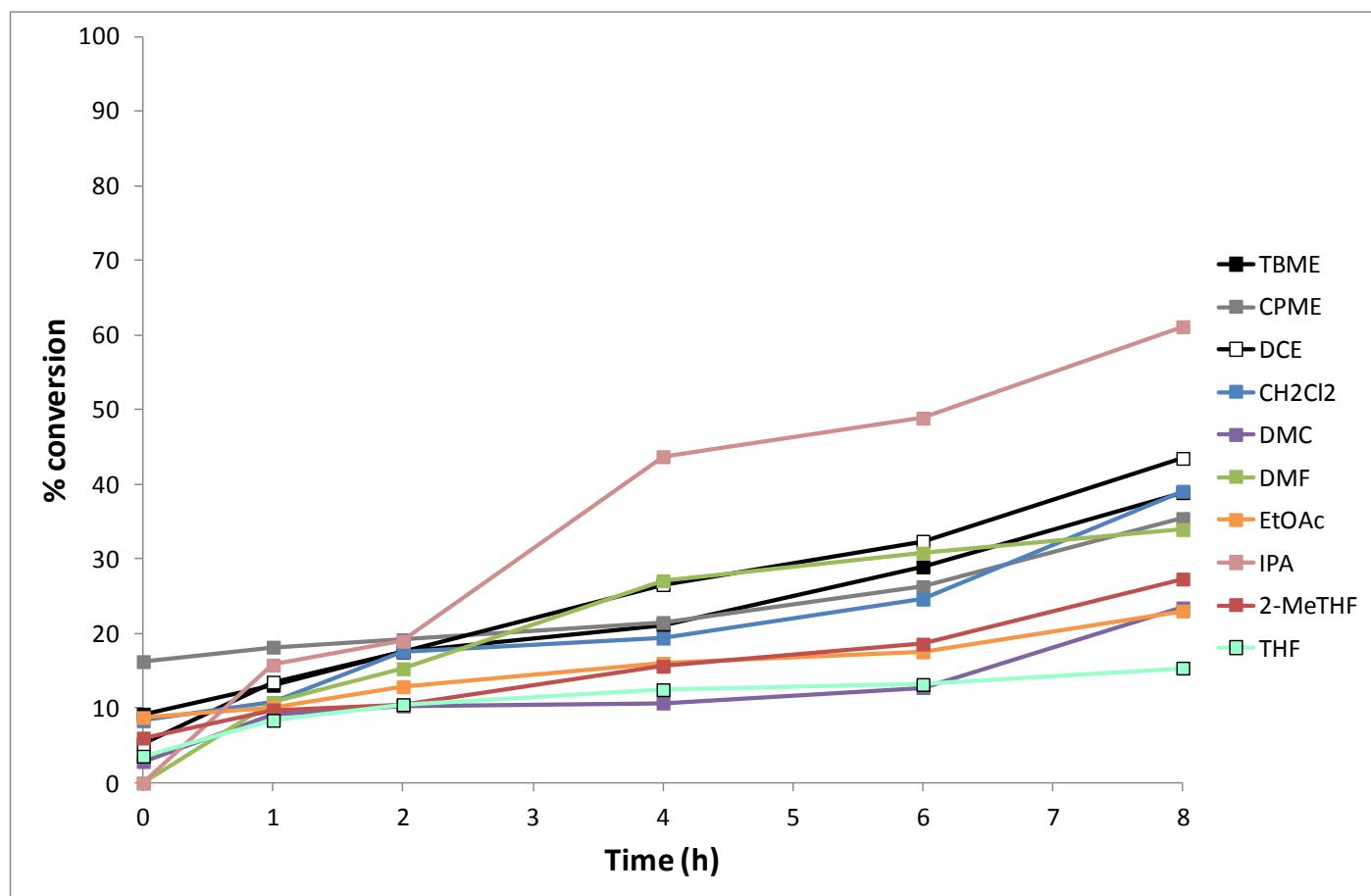


Time (h) / Solvent	TBME	CPME	DCE	CH ₂ Cl ₂	DMC	DMF	EtOAc	IPA	2-MeTHF	THF
0	19.5	35.8	20.8	67.4	37.7	8.5	3.1	33.4	5.9	8.4
1	21.7	41.7	65.2	71.8	43.5	56.1	3.5	46.0	8.4	10.4
2	21.7	41.7	66.0	76.5	43.5	58.3	3.5	46.0	8.4	10.4
4	21.7	41.7	67.2	78.2	43.5	58.7	3.5	46.0	8.4	10.4
6	21.7	41.7	67.3	79.8	43.5	59.3	3.6	46.0	8.4	10.4
8	21.7	41.7	70.0	80.8	43.5	60.2	4.0	46.0	8.4	10.4
24	21.7	41.7	73.2	82.5	43.5	93.6	31.0	46.0	8.4	10.4

Reaction 7: Sodium Triacetoxyborohydride

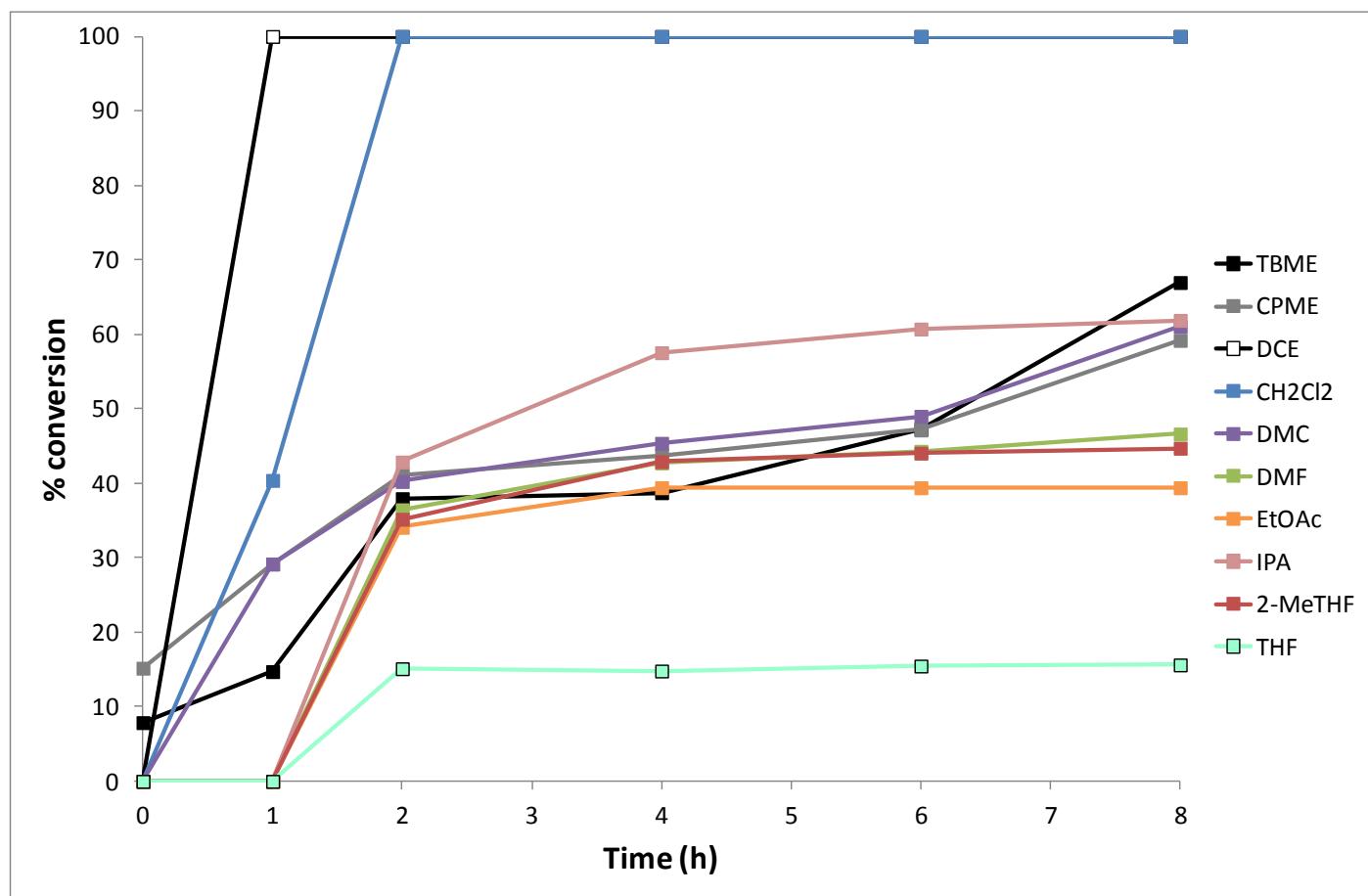


Reaction 7: Picoline-borane Complex



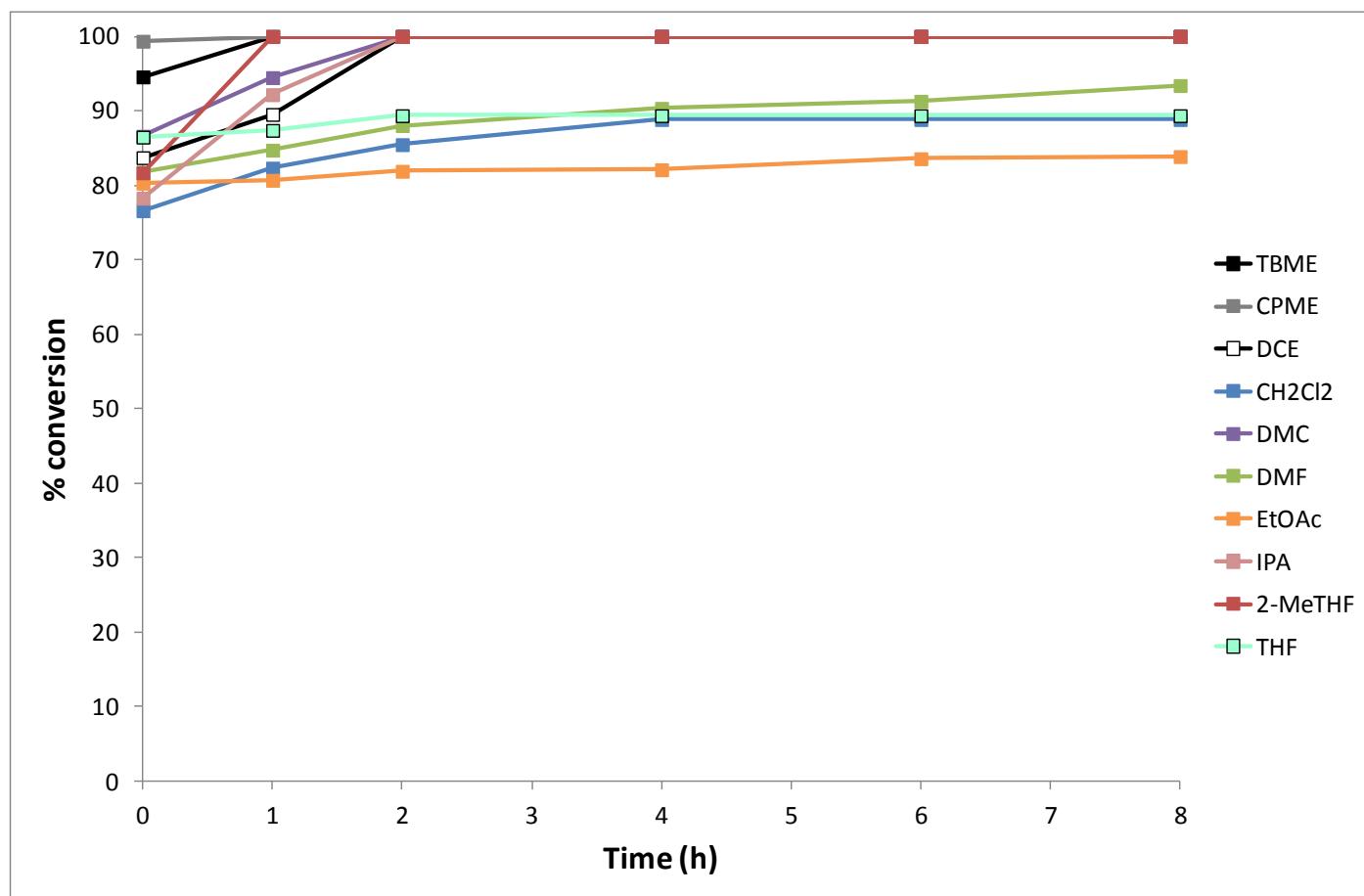
Time (h) / Solvent	TBME	CPME	DCE	CH ₂ Cl ₂	DMC	DMF	EtOAc	IPA	2-MeTHF	THF
0	9.2	16.3	5.2	8.3	2.9	0.0	8.8	0.0	6.0	3.6
1	13.1	18.2	13.5	10.8	9.1	10.8	10.1	15.8	9.8	8.4
2	17.6	19.2	17.6	17.6	10.3	15.3	12.9	19.0	10.4	10.5
4	21.2	21.5	26.6	19.4	10.7	27.1	16.0	43.7	15.6	15.3
6	29.0	26.3	32.4	24.6	12.8	30.8	17.6	48.9	18.6	15.3
8	38.9	35.4	43.5	39.1	23.5	34.0	23.0	61.1	27.3	15.3
24	59.3	55.4	58.0	60.8	41.7	66.5	46.2	74.0	65.8	27.9

Reaction 7: Sodium Cyanoborohydride



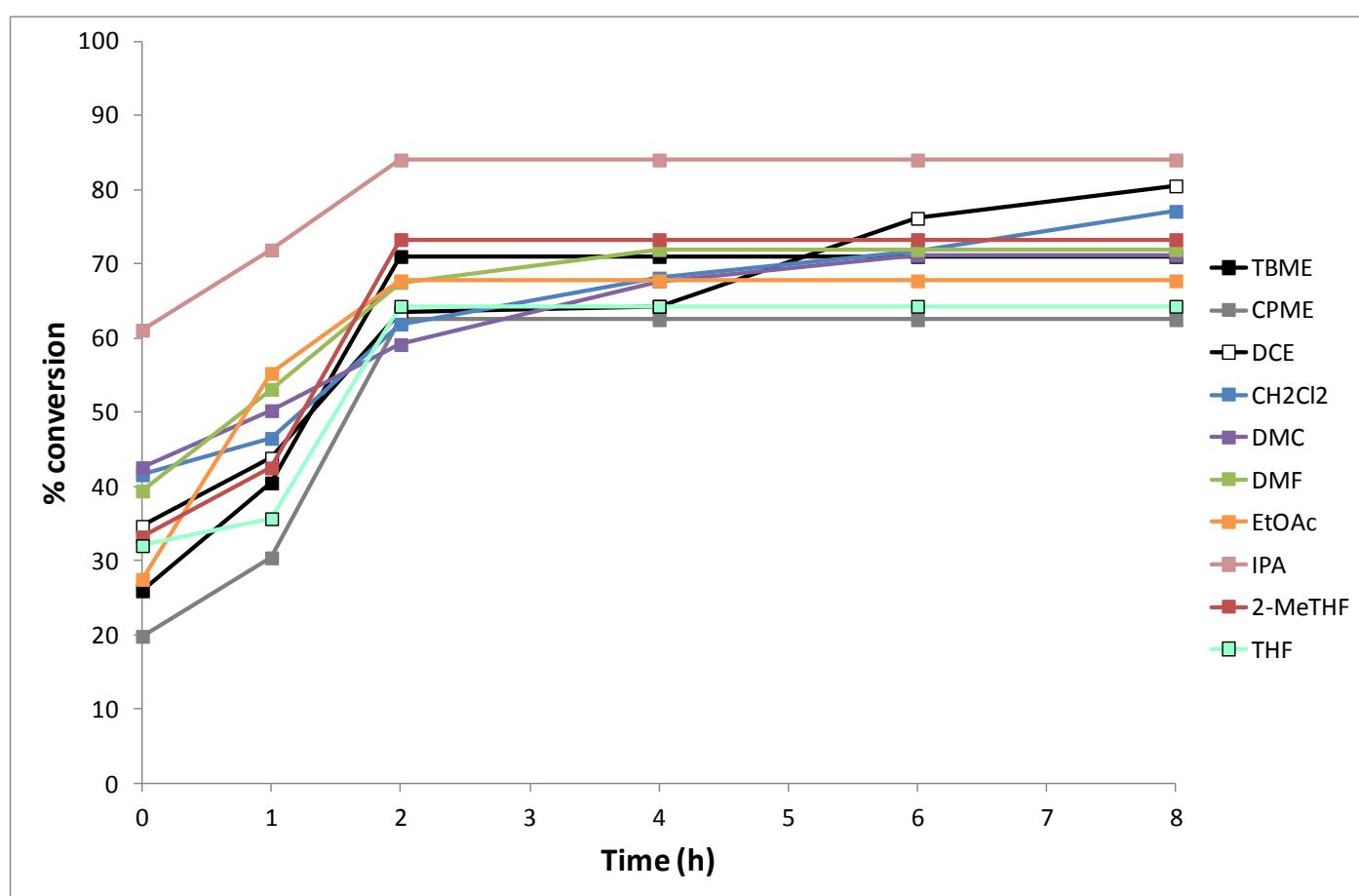
Time (h) / Solvent	TBME	CPME	DCE	CH ₂ Cl ₂	DMC	DMF	EtOAc	IPA	2-MeTHF	THF
0	7.9	15.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
1	14.7	29.2	100.0	40.4	29.2	0.0	0.0	0.0	0.0	0.0
2	37.9	41.1	100.0	100.0	40.3	36.5	34.1	42.9	35.2	15.1
4	38.7	43.8	100.0	100.0	45.4	42.7	39.4	57.5	42.9	14.8
6	47.3	47.2	100.0	100.0	49.0	44.3	39.7	60.7	44.1	15.5
8	67.0	59.2	100.0	100.0	61.1	46.6	25.9	61.9	44.7	15.6
24	82.0	59.2	100.0	100.0	100.0	46.6	37.8	68.1	44.7	22.2

Reaction 8: Sodium Triacetoxyborohydride



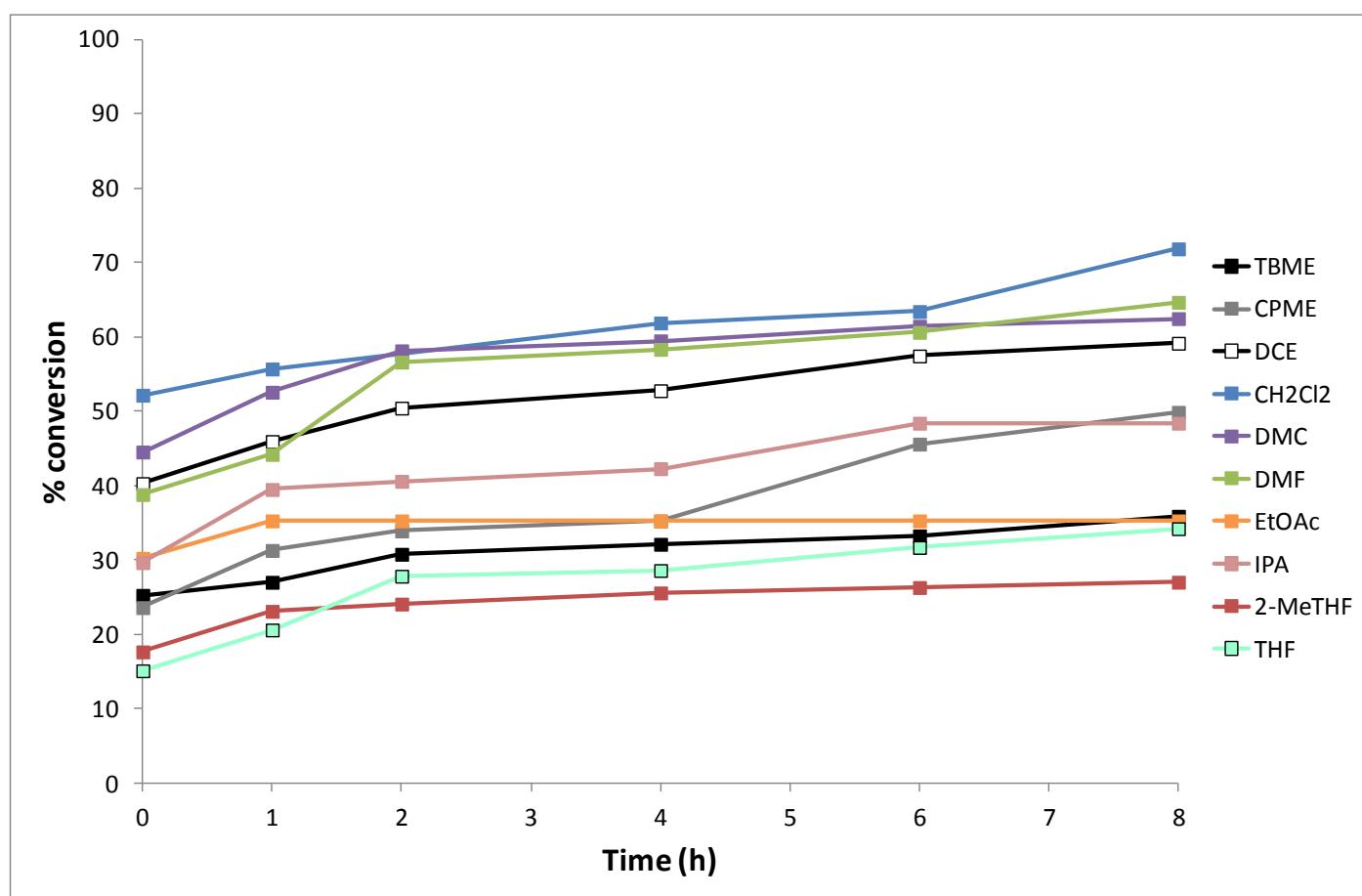
Time (h) / Solvent	TBME	CPME	DCE	CH ₂ Cl ₂	DMC	DMF	EtOAc	IPA	2-MeTHF	THF
0	94.6	99.4	83.7	76.6	86.6	81.8	80.3	78.3	81.7	86.5
1	100.0	100.0	89.5	82.3	94.5	84.7	80.7	92.2	100.0	87.4
2	100.0	100.0	100.0	85.5	100.0	88.0	81.9	100.0	100.0	89.4
4	100.0	100.0	100.0	88.8	100.0	90.4	82.1	100.0	100.0	89.4
6	100.0	100.0	100.0	88.8	100.0	91.3	83.6	100.0	100.0	89.4
8	100.0	100.0	100.0	88.8	100.0	93.4	83.9	100.0	100.0	89.4
24	100.0	100.0	100.0	88.8	100.0	100.0	93.7	100.0	100.0	89.4

Reaction 8: Picoline-borane Complex



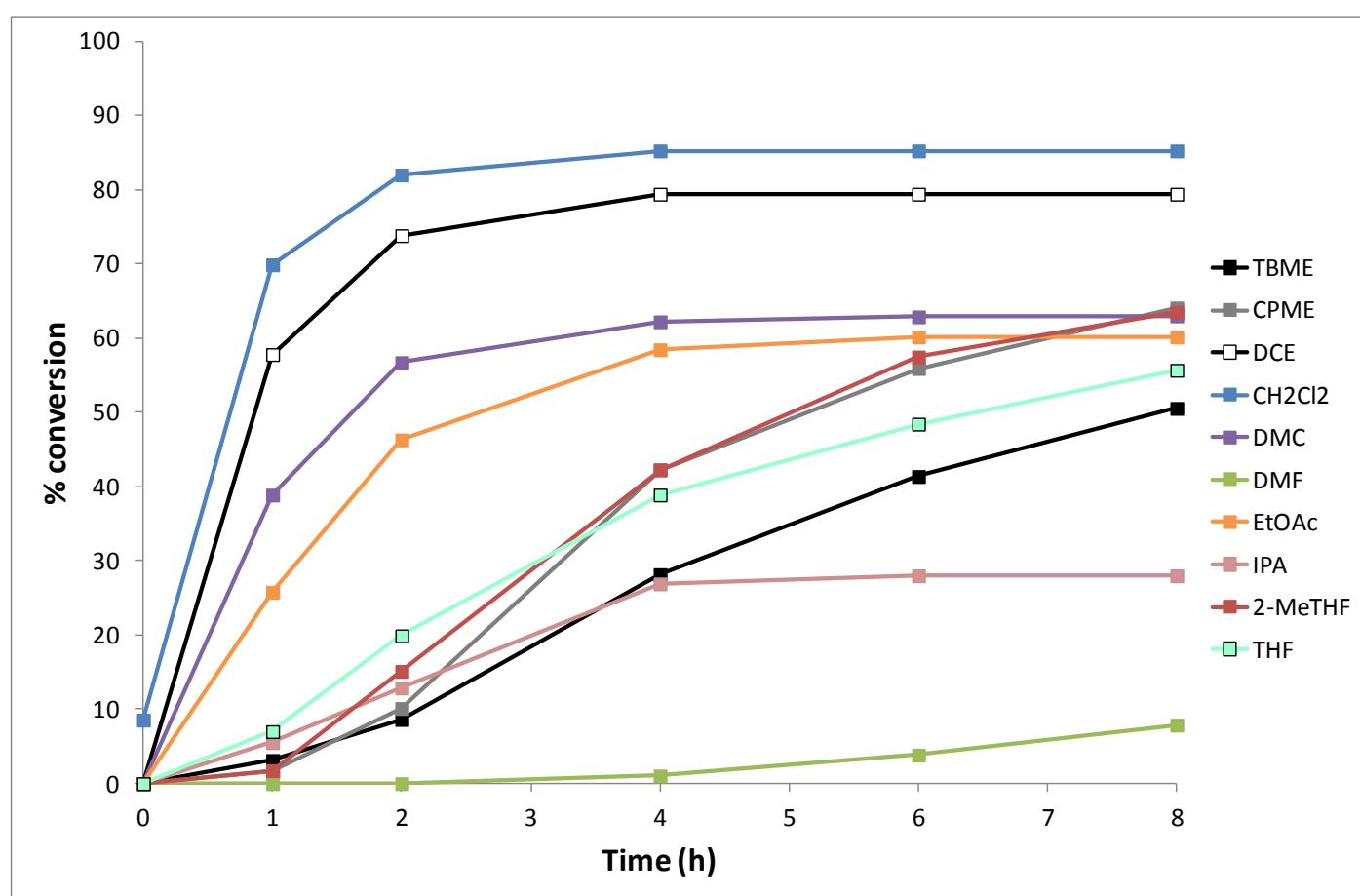
Time (h) / Solvent	TBME	CPME	DCE	CH ₂ Cl ₂	DMC	DMF	EtOAc	IPA	2-MeTHF	THF
0	26.0	19.8	34.7	41.6	42.6	39.4	27.5	61.1	33.2	32.0
1	40.5	30.4	43.8	46.5	50.2	53.1	55.3	71.9	42.5	35.7
2	71.0	62.5	63.5	61.9	59.1	67.5	67.7	84.0	73.3	64.3
4	71.0	62.5	64.3	68.1	67.7	71.9	67.7	84.0	73.3	64.3
6	71.0	62.5	76.1	71.6	71.2	71.9	67.7	84.0	73.3	64.3
8	71.0	62.5	80.5	77.1	71.2	71.9	67.7	84.0	73.3	64.3
24	71.0	62.5	81.4	82.4	71.2	71.9	67.7	84.0	73.3	64.3

Reaction 8: Sodium Cyanoborohydride



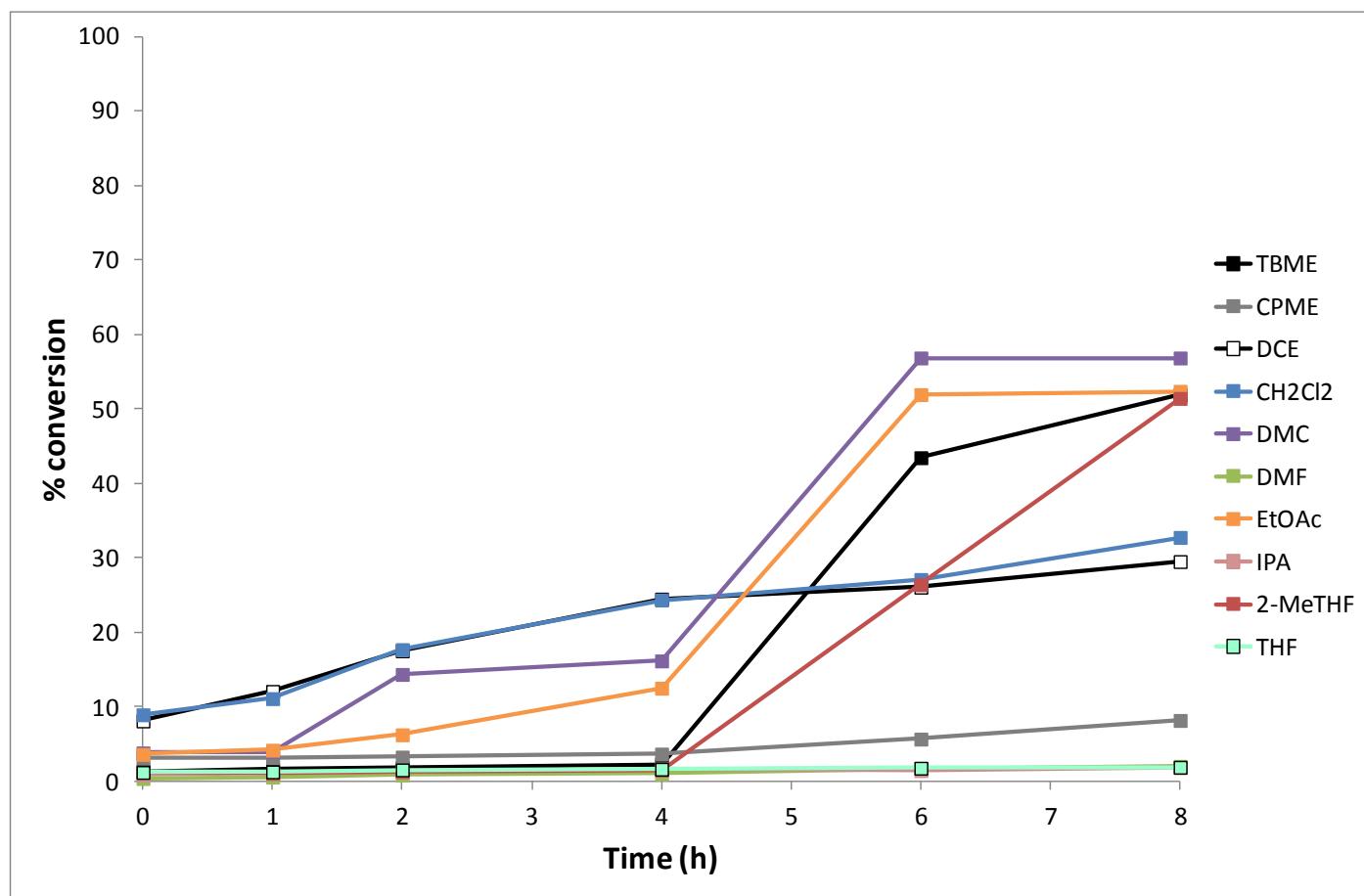
Time (h) / Solvent	TBME	CPME	DCE	CH ₂ Cl ₂	DMC	DMF	EtOAc	IPA	2-MeTHF	THF
0	25.3	23.6	40.3	52.2	44.5	38.8	30.2	29.7	17.7	15.1
1	27.0	31.3	46.0	55.7	52.6	44.2	35.3	39.5	23.1	20.6
2	30.8	33.9	50.4	57.6	58.1	56.6	35.3	40.6	24.1	27.8
4	32.1	35.3	52.8	61.9	59.5	58.3	35.3	42.3	25.6	28.6
6	33.3	45.6	57.4	63.4	61.4	60.6	35.3	48.4	26.3	31.7
8	35.9	49.9	59.2	71.8	62.5	64.7	35.3	48.4	27.0	34.2
24	35.9	51.2	59.2	72.4	62.5	64.7	35.3	48.4	27.0	34.7

Reaction 9: Sodium Triacetoxyborohydride



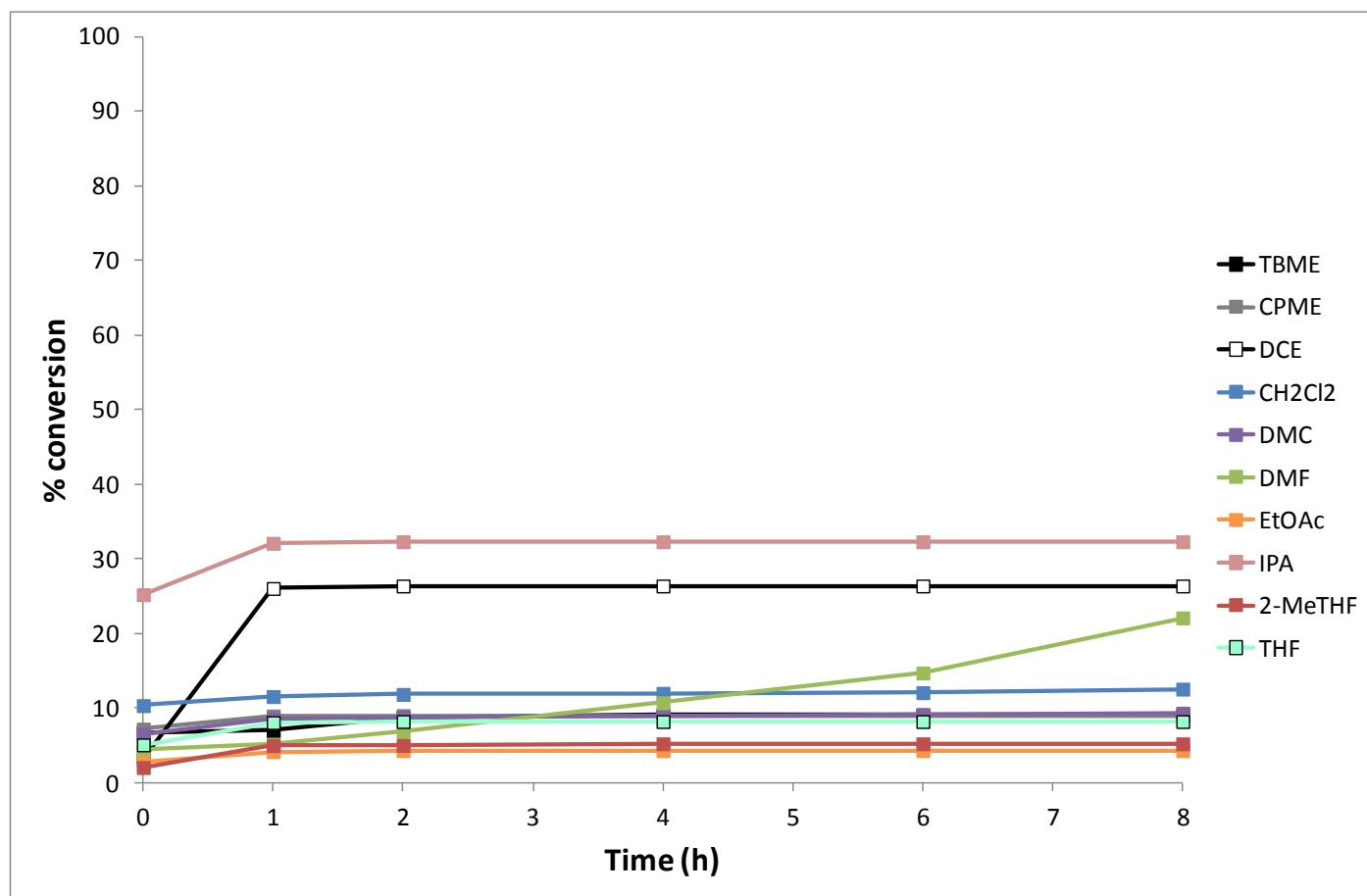
Time (h) / Solvent	TBME	CPME	DCE	CH ₂ Cl ₂	DMC	DMF	EtOAc	IPA	2-MeTHF	THF
0	0.0	0.0	0.0	8.6	0.0	0.0	0.0	0.0	0.0	0.0
1	3.1	1.7	57.8	69.9	38.9	0.0	25.8	5.5	1.7	7.0
2	8.6	10.1	73.8	82.0	56.7	0.0	46.3	12.9	15.1	19.9
4	28.1	42.3	79.4	85.2	62.2	1.0	58.4	26.9	42.3	38.9
6	41.4	55.9	79.4	85.2	62.9	3.8	60.2	28.0	57.5	48.4
8	50.5	64.1	79.4	85.2	63.0	7.9	60.2	28.0	63.5	55.7
24	73.8	81.4	79.4	85.2	63.7	32.5	60.2	28.0	76.6	69.1

Reaction 9: Picoline-borane Complex



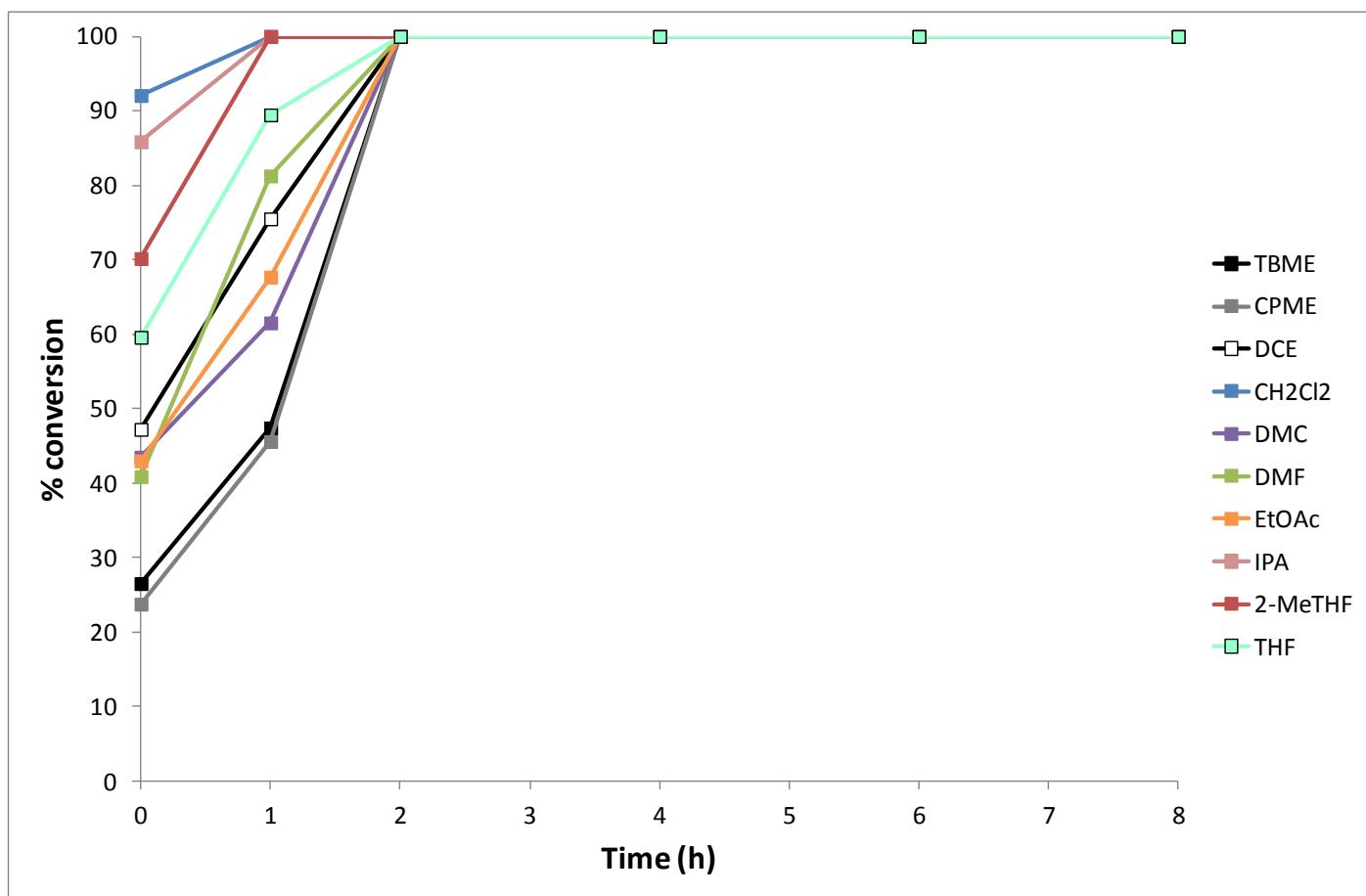
Time (h) / Solvent	TBME	CPME	DCE	CH ₂ Cl ₂	DMC	DMF	EtOAc	IPA	2-MeTHF	THF
0	1.4	3.1	8.1	9.0	3.8	0.4	3.6	0.9	1.2	1.2
1	1.7	3.2	12.1	11.1	4.0	0.6	4.2	1.1	1.1	1.3
2	1.9	3.3	17.5	17.7	14.4	0.9	6.3	1.3	1.3	1.6
4	2.3	3.7	24.5	24.4	16.2	1.0	12.5	1.7	1.5	1.7
6	43.5	5.7	26.1	27.1	56.9	1.8	51.9	1.5	26.4	1.8
8	51.9	8.2	29.5	32.8	56.9	2.0	52.4	1.9	51.4	1.9
24	51.9	51.3	61.4	69.7	56.9	59.4	52.4	71.4	51.4	52.6

Reaction 9: Sodium Cyanoborohydride

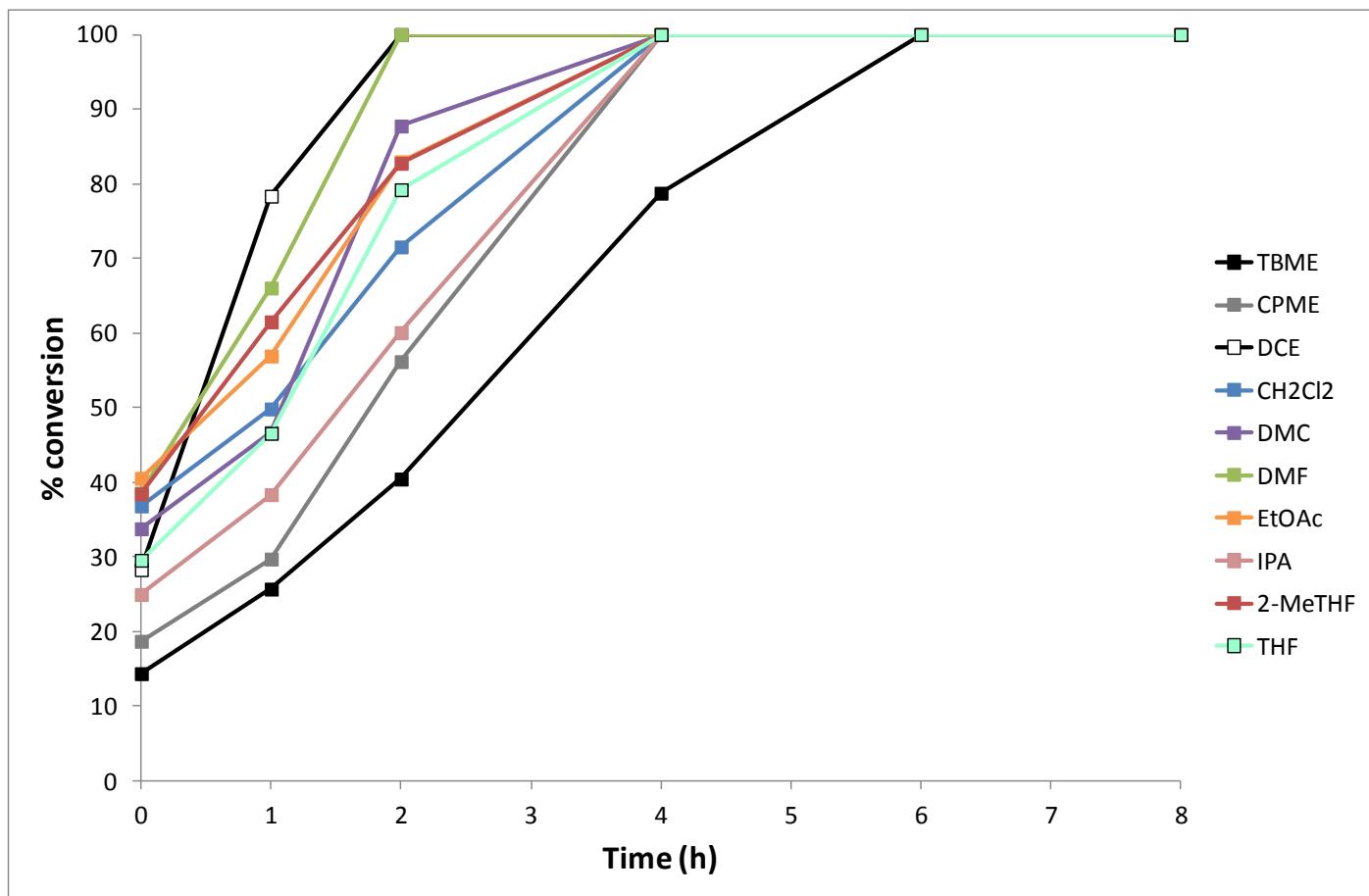


Time (h) / Solvent	TBME	CPME	DCE	CH ₂ Cl ₂	DMC	DMF	EtOAc	IPA	2-MeTHF	THF
0	6.8	7.2	3.2	10.4	6.5	4.5	2.8	25.2	2.1	5.1
1	7.0	9.0	26.1	11.6	8.6	5.2	4.2	32.1	5.0	8.1
2	8.8	9.0	26.4	11.9	8.8	6.8	4.3	32.3	5.0	8.2
4	9.1	9.0	26.4	12.0	8.9	10.7	4.3	32.3	5.3	8.2
6	9.1	9.0	26.4	12.1	9.1	14.7	4.3	32.3	5.3	8.2
8	9.1	9.0	26.4	12.6	9.4	22.1	4.3	32.3	5.3	8.2
24	9.1	9.0	26.4	12.6	9.4	22.1	4.3	32.3	5.3	8.2

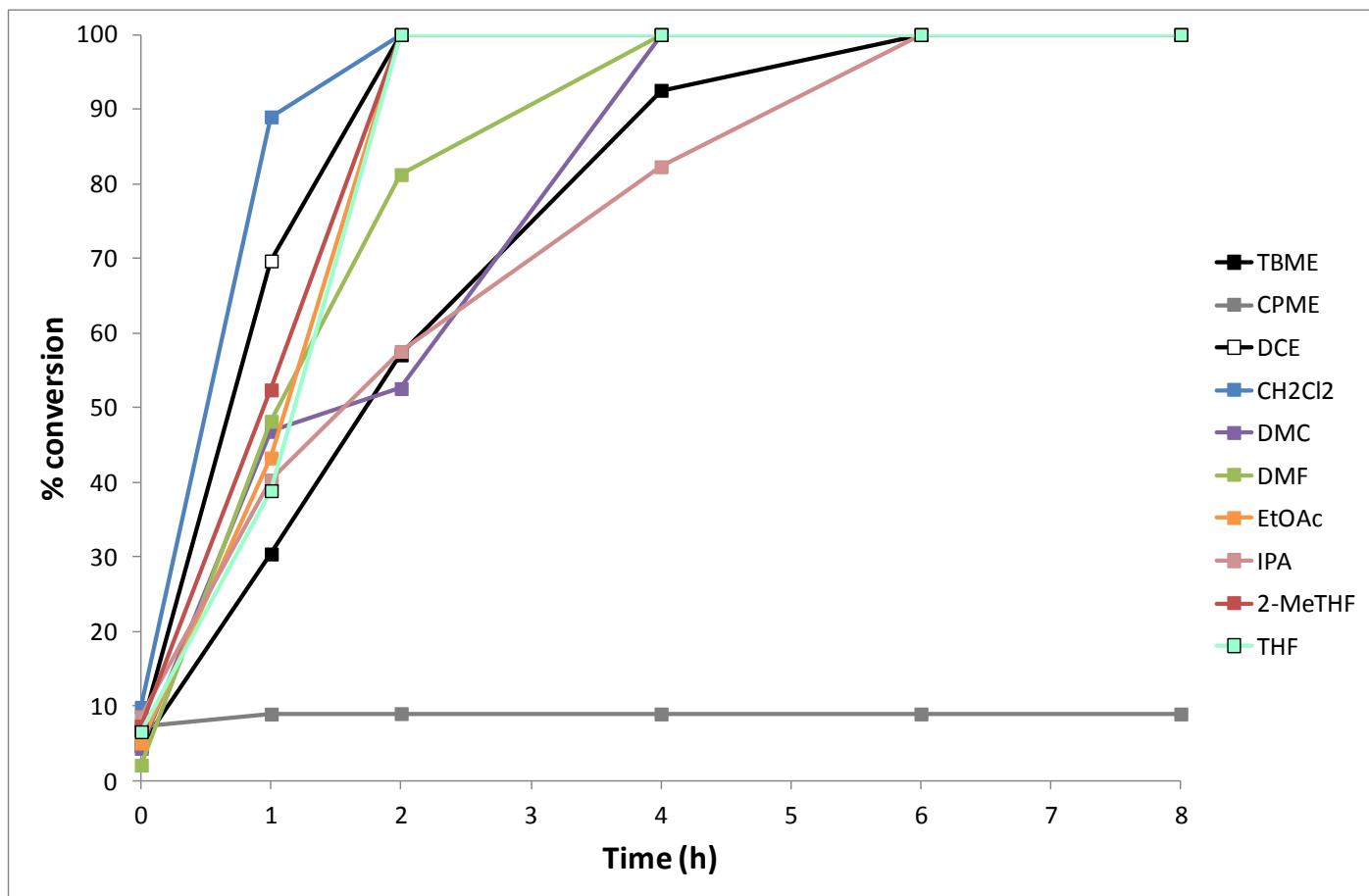
Reaction 10: Sodium Triacetoxyborohydride



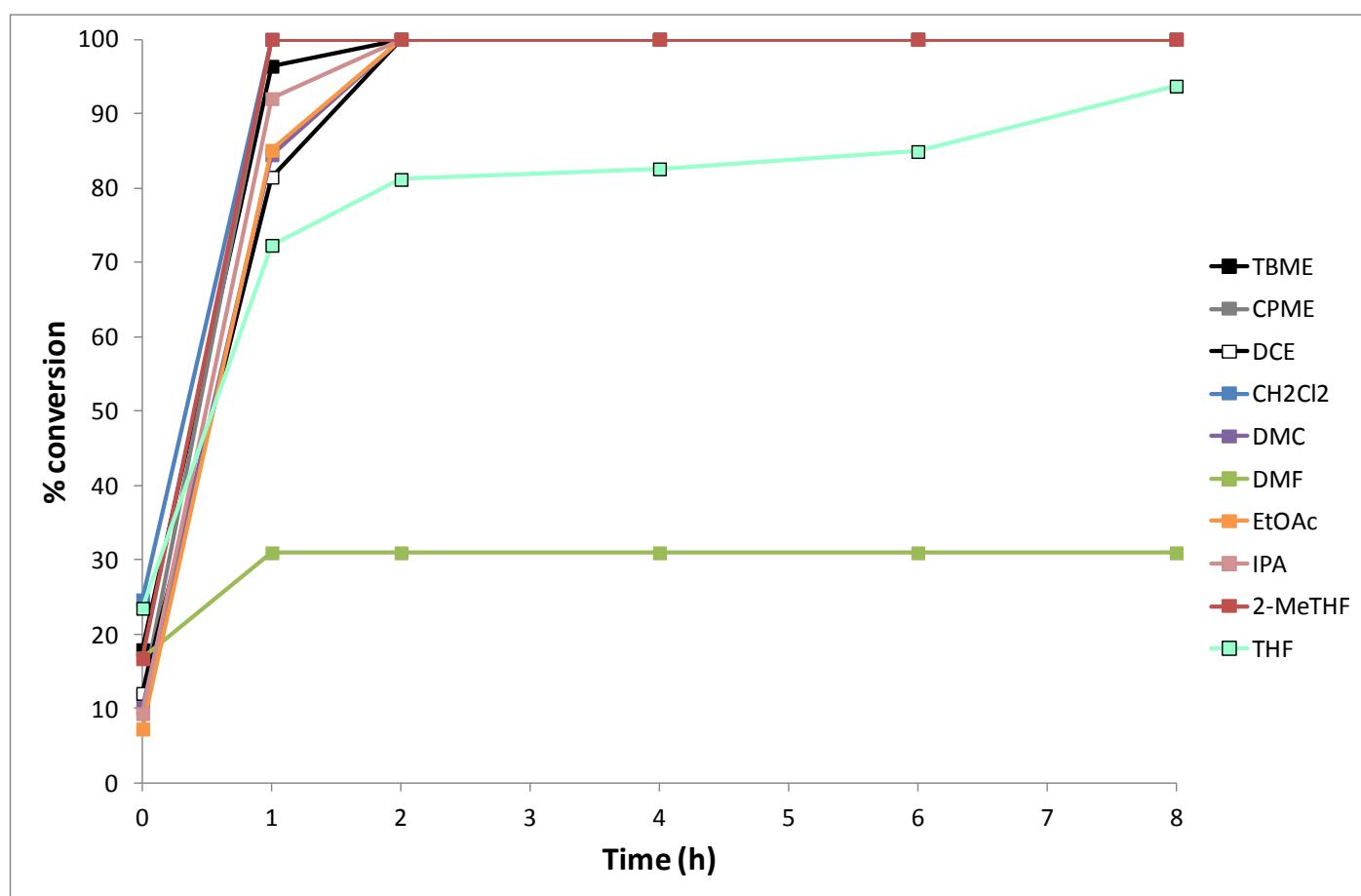
Reaction 10: Picoline-borane Complex



Reaction 10: Sodium Cyanoborohydride

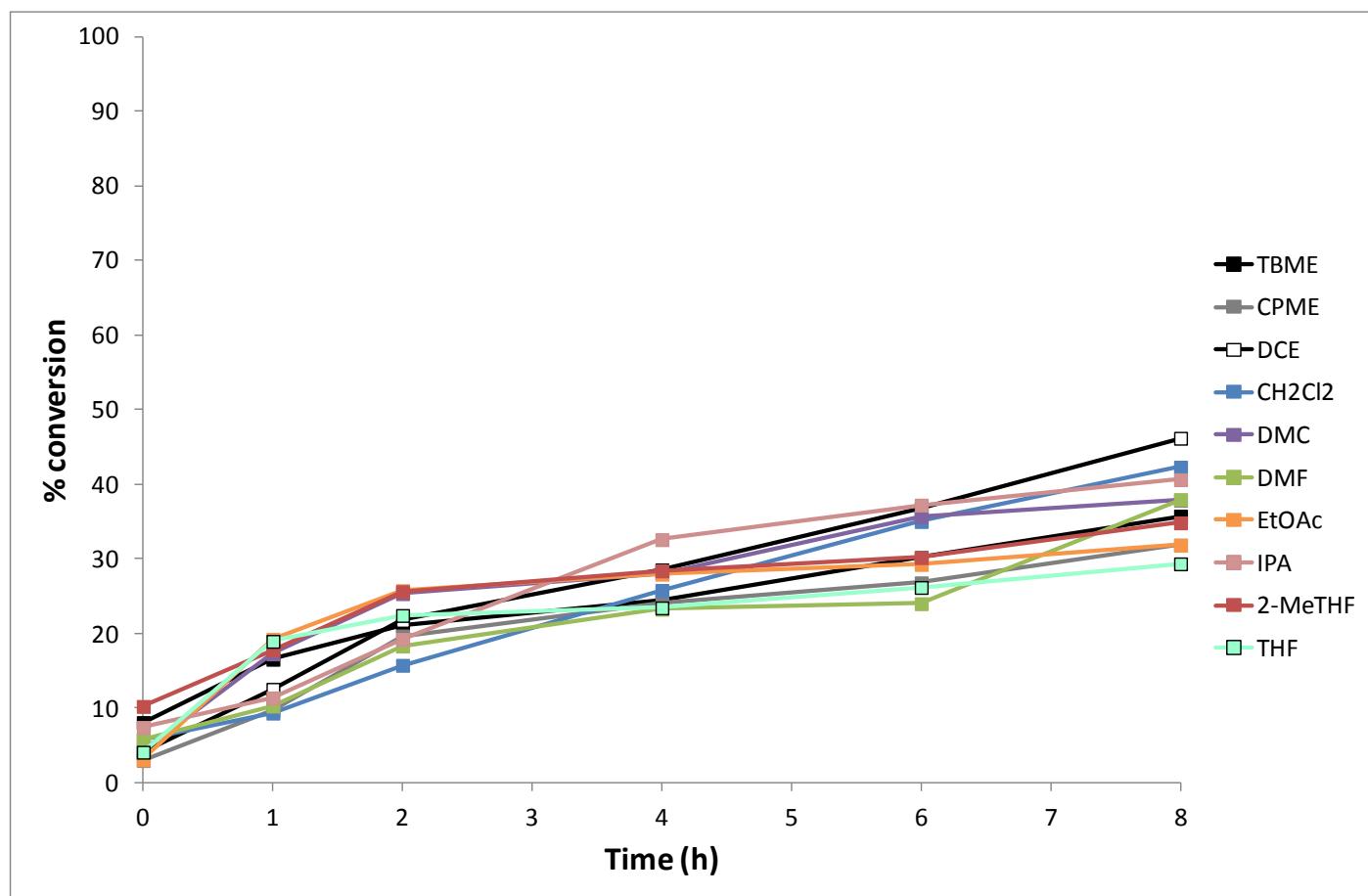


Reaction 11: Sodium Triacetoxyborohydride



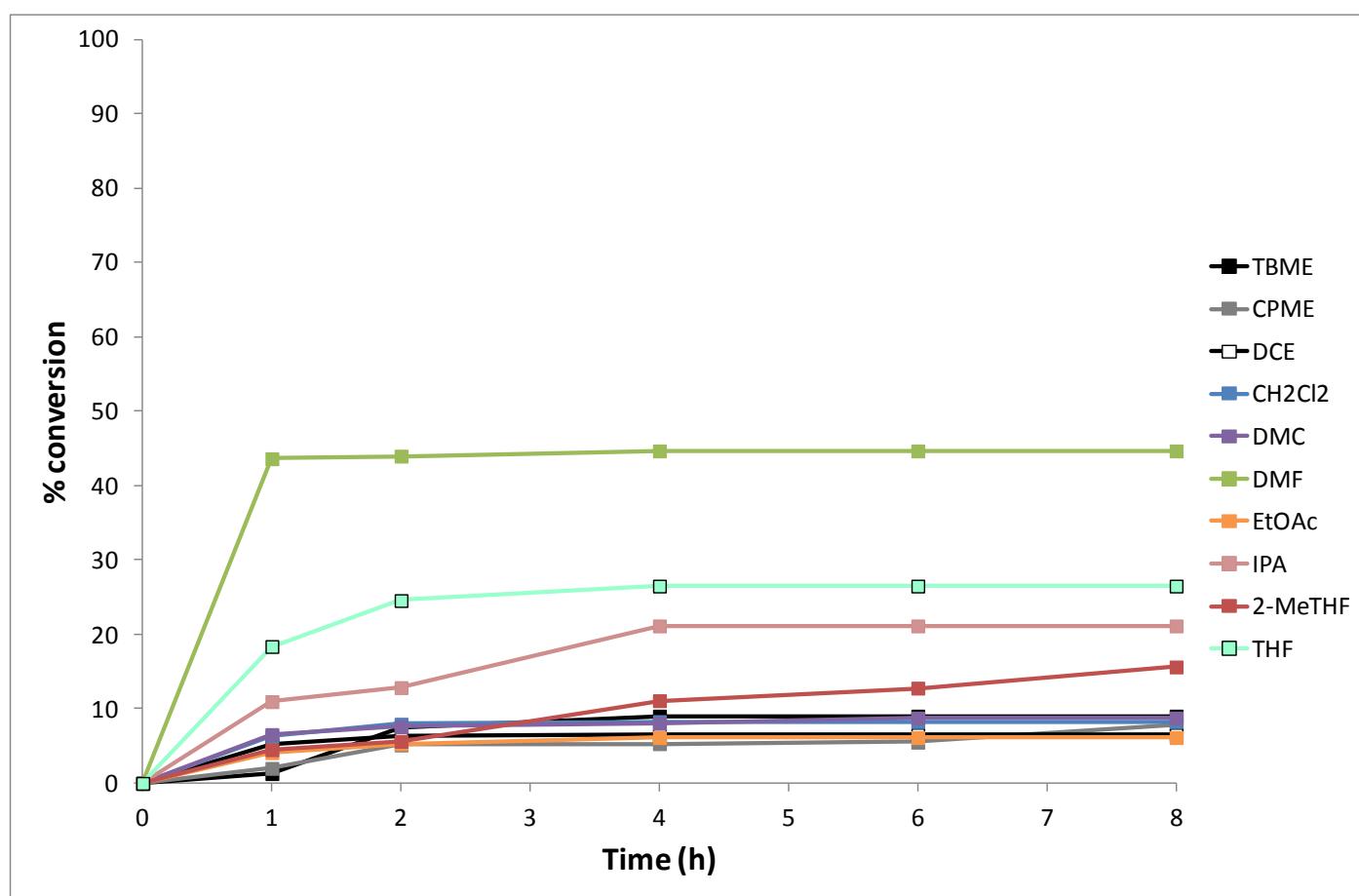
Time (h) / Solvent	TBME	CPME	DCE	CH ₂ Cl ₂	DMC	DMF	EtOAc	IPA	2-MeTHF	THF
0	18.0	11.7	12.1	24.7	10.1	16.8	7.3	9.4	16.7	23.5
1	96.4	100.0	81.5	100.0	84.5	31.0	85.1	92.0	100.0	72.3
2	100.0	100.0	100.0	100.0	100.0	31.0	100.0	100.0	100.0	81.2
4	100.0	100.0	100.0	100.0	100.0	31.0	100.0	100.0	100.0	82.6
6	100.0	100.0	100.0	100.0	100.0	31.0	100.0	100.0	100.0	84.9
8	100.0	100.0	100.0	100.0	100.0	31.0	100.0	100.0	100.0	93.7
24	100.0	100.0	100.0	100.0	100.0	31.0	100.0	100.0	100.0	100.0

Reaction 11: Picoline-borane Complex



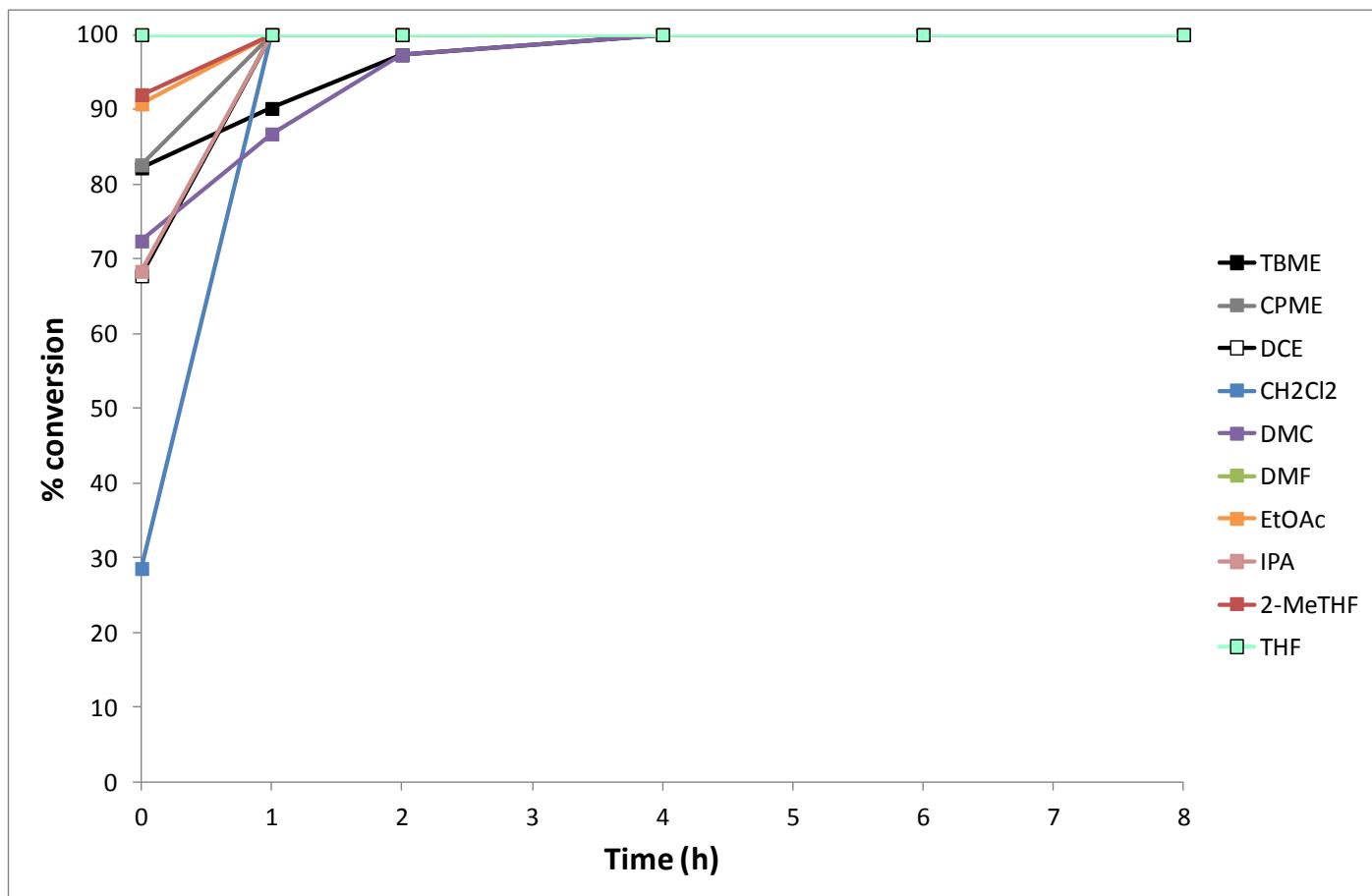
Time (h) / Solvent	TBME	CPME	DCE	CH ₂ Cl ₂	DMC	DMF	EtOAc	IPA	2-MeTHF	THF
0	8.1	3.0	4.0	5.8	4.4	5.7	3.1	7.4	10.3	4.1
1	16.6	9.8	12.5	9.4	17.3	10.3	19.2	11.4	17.8	19.0
2	21.0	19.7	21.8	15.8	25.4	18.3	25.8	19.2	25.6	22.4
4	24.5	24.1	28.5	25.8	28.1	23.3	28.0	32.6	28.4	23.5
6	30.2	26.8	36.7	35.0	35.7	24.0	29.3	37.2	30.2	26.2
8	35.8	31.9	46.2	42.3	37.9	38.0	31.9	40.6	34.9	29.4
24	40.8	38.4	61.1	63.5	44.7	63.9	45.3	40.6	43.2	36.9

Reaction 11: Sodium Cyanoborohydride

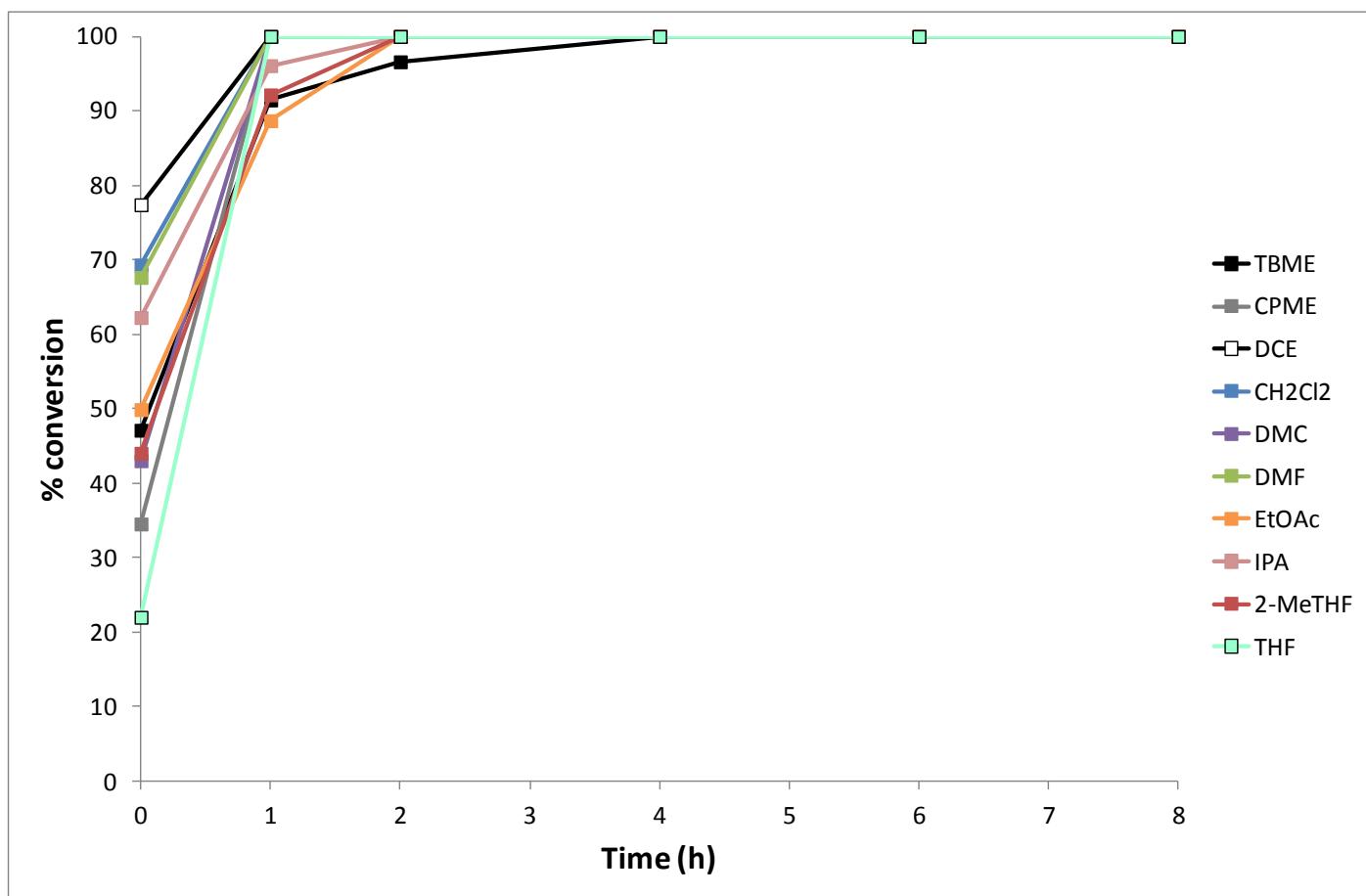


Time (h) / Solvent	TBME	CPME	DCE	CH ₂ Cl ₂	DMC	DMF	EtOAc	IPA	2-MeTHF	THF
0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
1	1.3	2.0	5.2	6.3	6.6	43.6	4.1	11.0	4.5	18.4
2	7.5	5.2	6.3	8.0	7.6	44.0	5.3	12.9	5.6	24.6
4	9.0	5.3	6.5	8.3	8.1	44.7	6.2	21.2	11.1	26.5
6	9.0	5.5	6.5	8.3	8.8	44.7	6.2	21.2	12.8	26.5
8	9.0	7.9	6.5	8.3	8.8	44.7	6.2	21.2	15.6	26.5
24	9.0	10.5	6.5	8.3	8.8	44.7	6.2	21.2	40.5	26.5

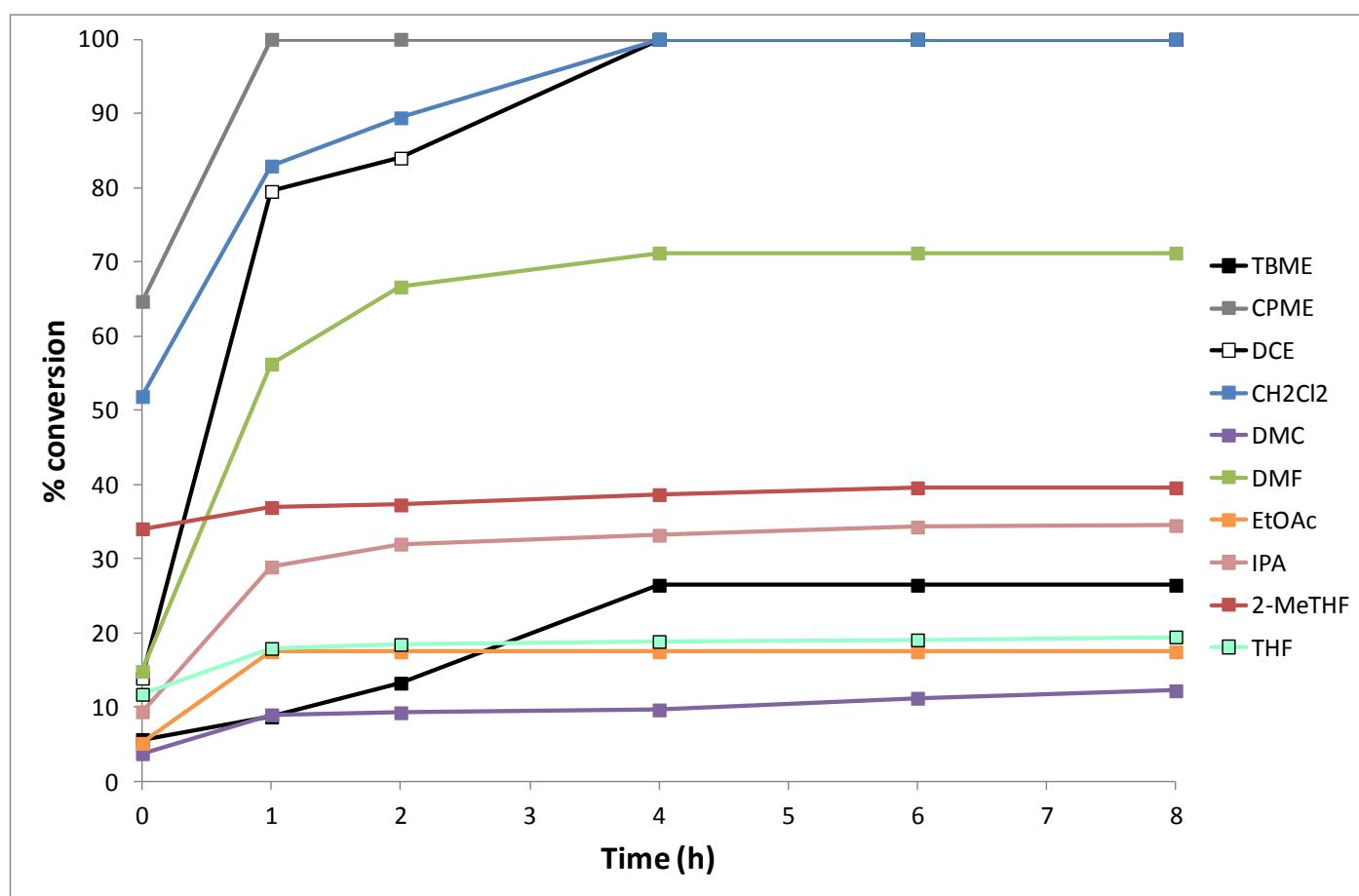
Reaction 12: Sodium Triacetoxyborohydride



Reaction 12: Picoline-borane Complex



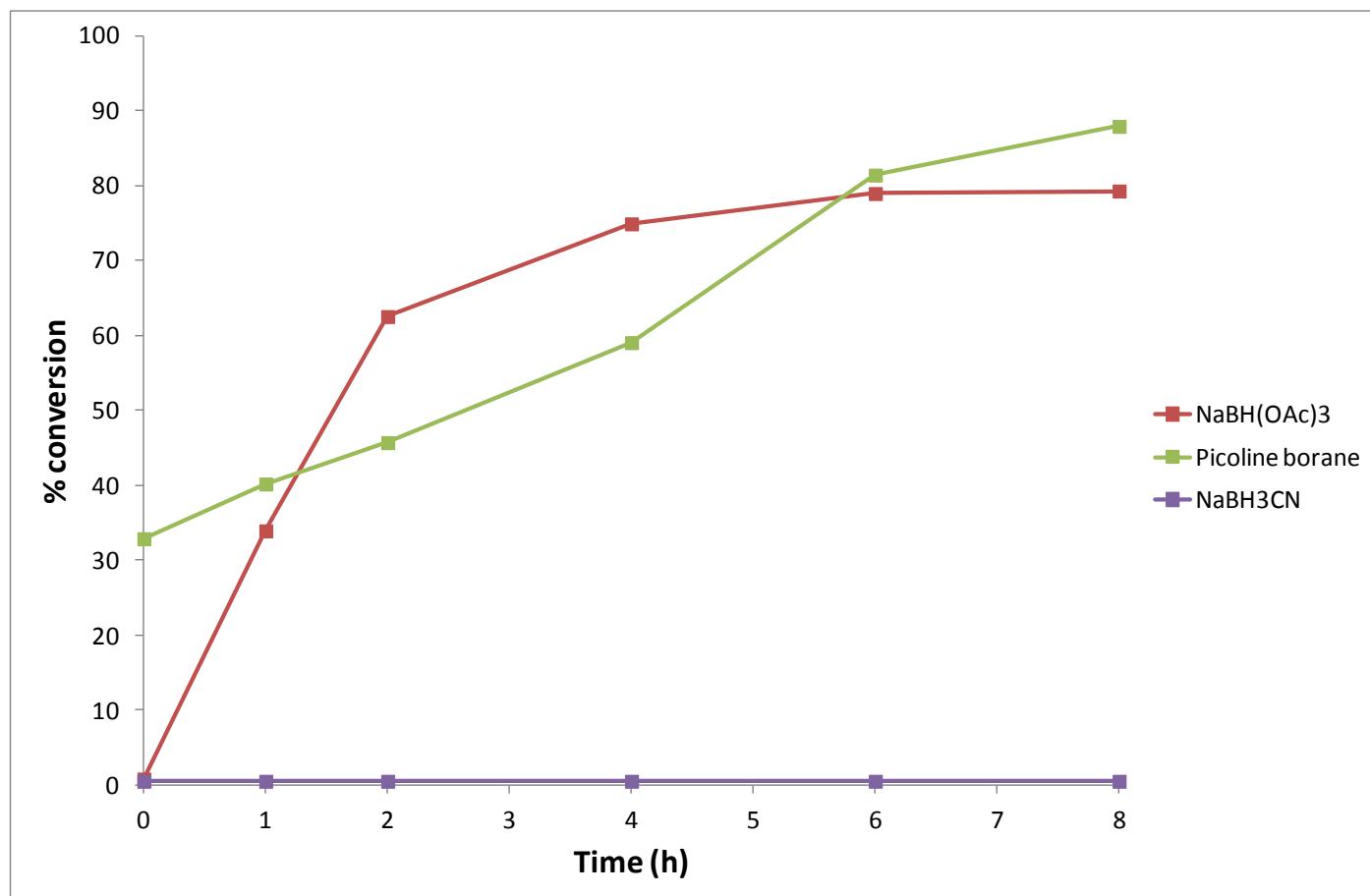
Reaction 12: Sodium Cyanoborohydride



Time (h) / Solvent	TBME	CPME	DCE	CH ₂ Cl ₂	DMC	DMF	EtOAc	IPA	2-MeTHF	THF
0	5.7	64.7	13.9	51.9	3.8	14.9	5.2	9.4	34.1	11.8
1	8.7	100.0	79.5	82.9	9.0	56.3	17.5	28.9	36.9	17.9
2	13.3	100.0	84.0	89.4	9.3	66.6	17.5	32.0	37.3	18.5
4	26.5	100.0	100.0	100.0	9.7	71.2	17.5	33.2	38.7	18.9
6	26.5	100.0	100.0	100.0	11.2	71.2	17.5	34.3	39.6	19.1
8	26.5	100.0	100.0	100.0	12.2	71.2	17.5	34.5	39.6	19.5
24	26.5	100.0	100.0	100.0	12.7	71.2	17.5	37.1	39.6	20.4

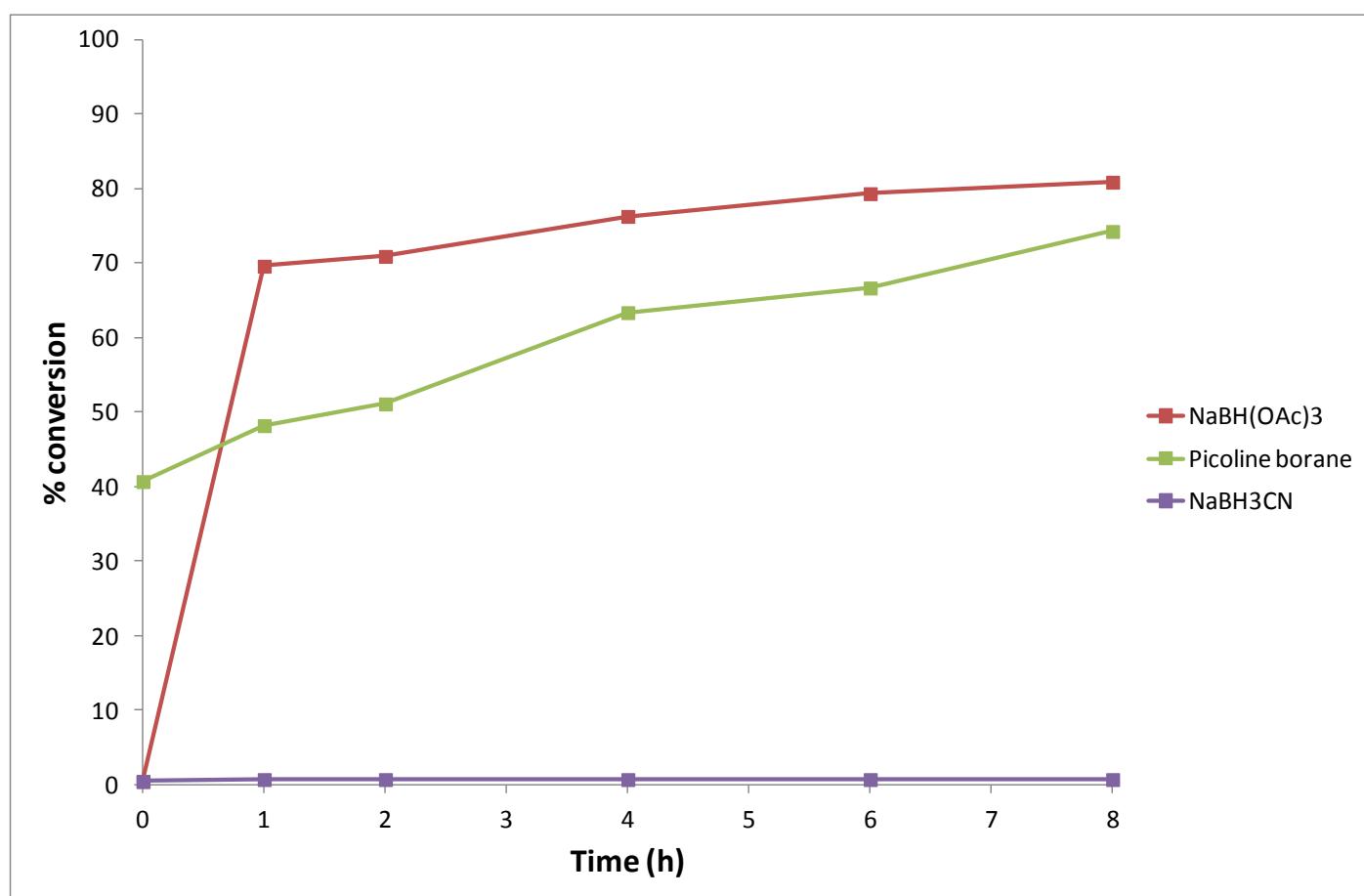
3.2 Conversion vs. Time Data for Reactions 1-12 Using the Range of Reducing Agents in a Specific Solvent

Reaction 1: TBME



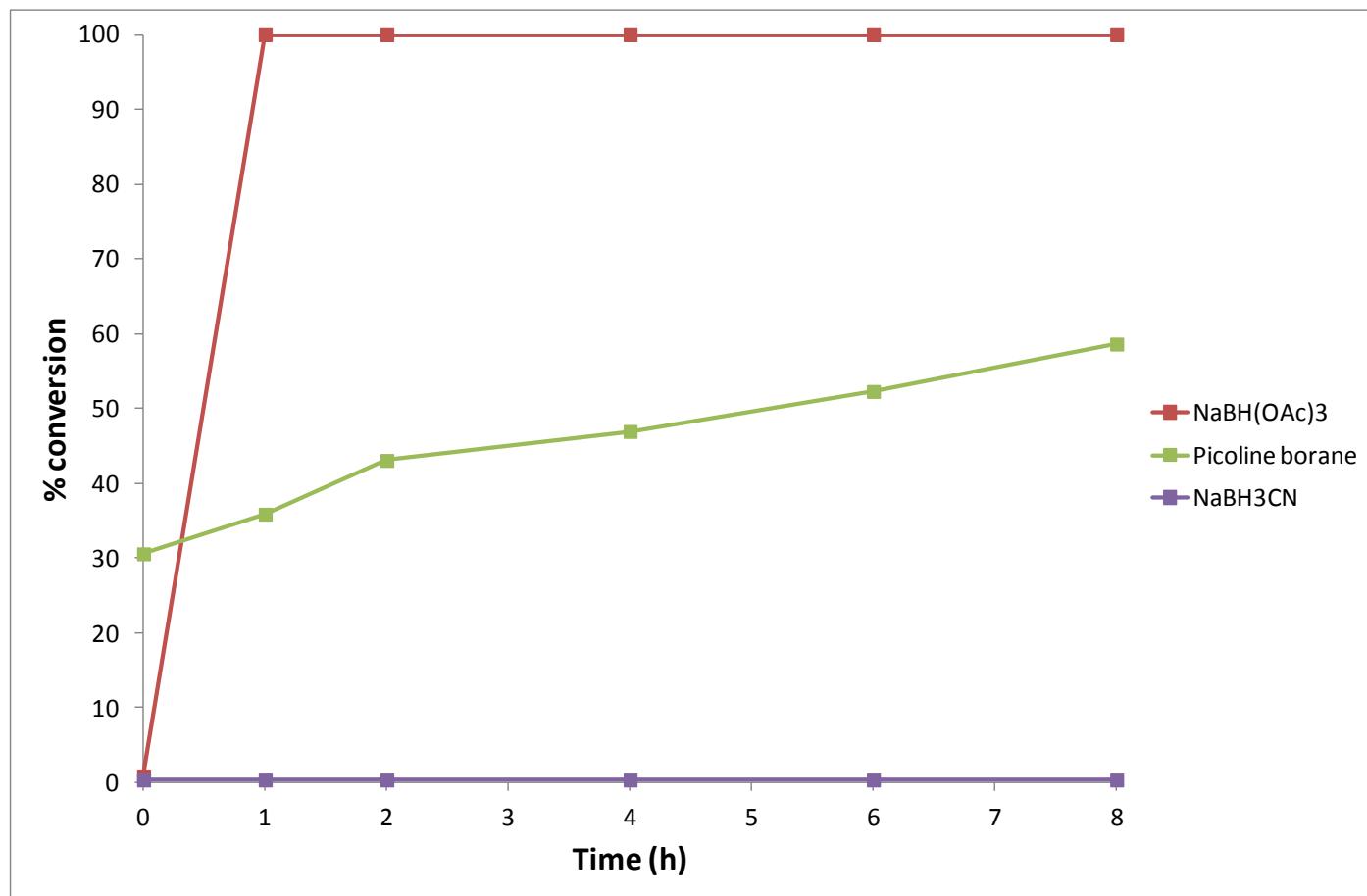
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	0.8	32.9	0.5
1	33.9	40.2	0.5
2	62.5	45.7	0.5
4	74.9	59.1	0.5
6	78.9	81.4	0.5
8	79.3	87.9	0.5
24	84.5	89.7	0.5

Reaction 1: CPME



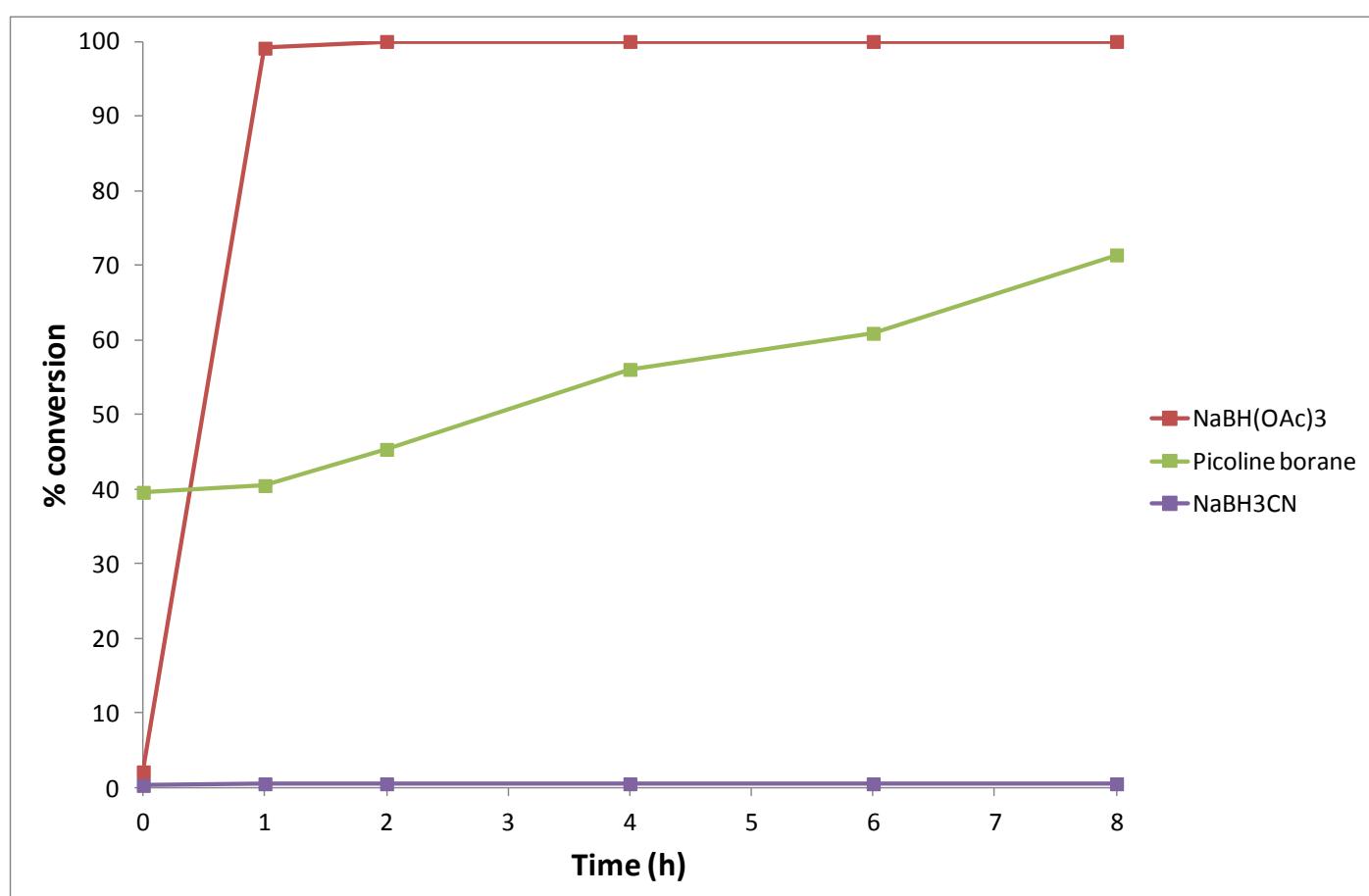
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	0.5	40.7	0.5
1	69.6	48.2	0.7
2	70.9	51.1	0.7
4	76.3	63.4	0.7
6	79.3	66.7	0.7
8	80.8	74.2	0.7
24	85.6	80.2	0.7

Reaction 1: DCE



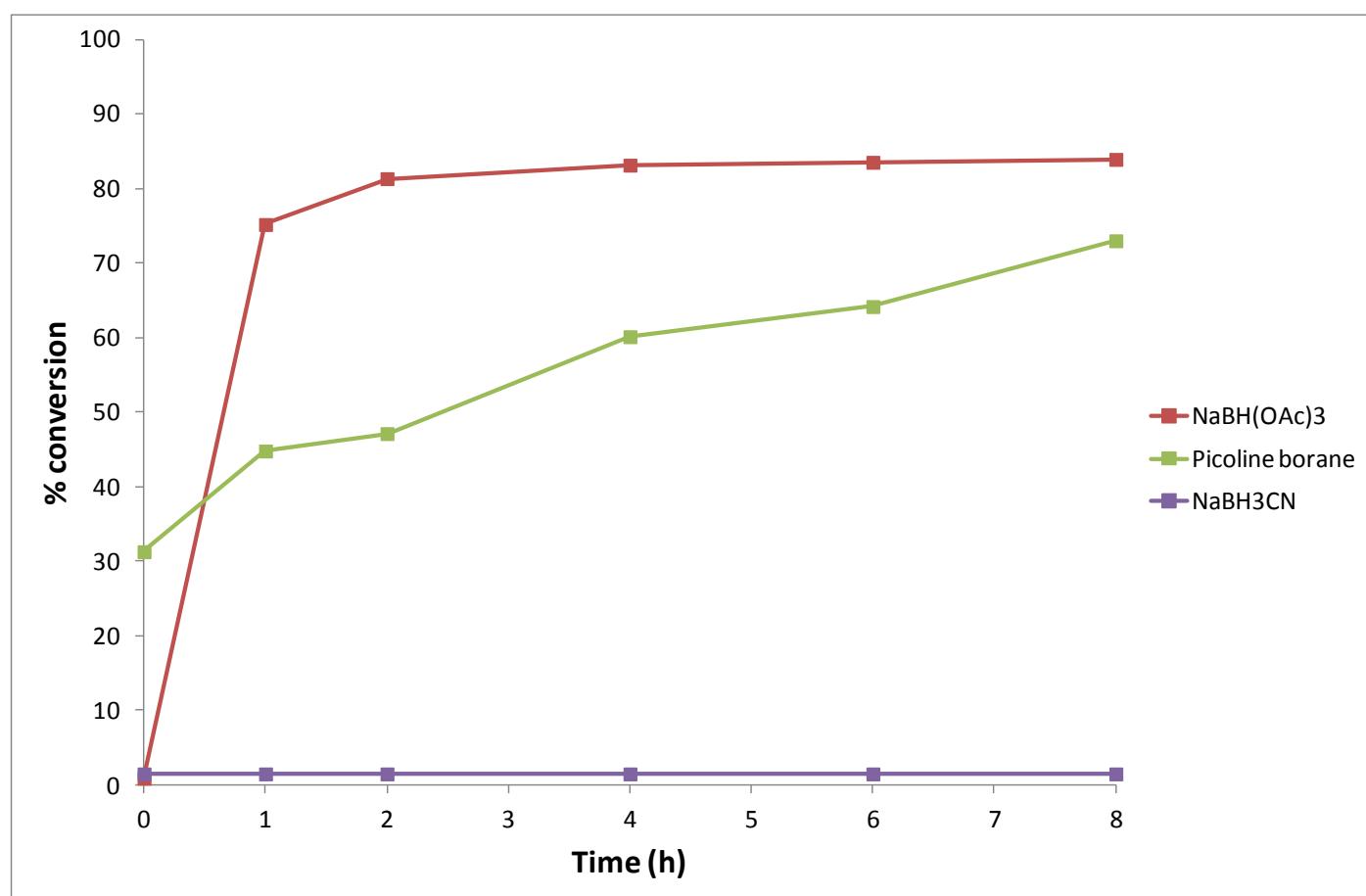
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	0.9	30.6	0.4
1	100.0	35.9	0.4
2	100.0	43.1	0.4
4	100.0	47.0	0.4
6	100.0	52.3	0.4
8	100.0	58.6	0.4
24	100.0	65.0	0.4

Reaction 1: CH₂Cl₂



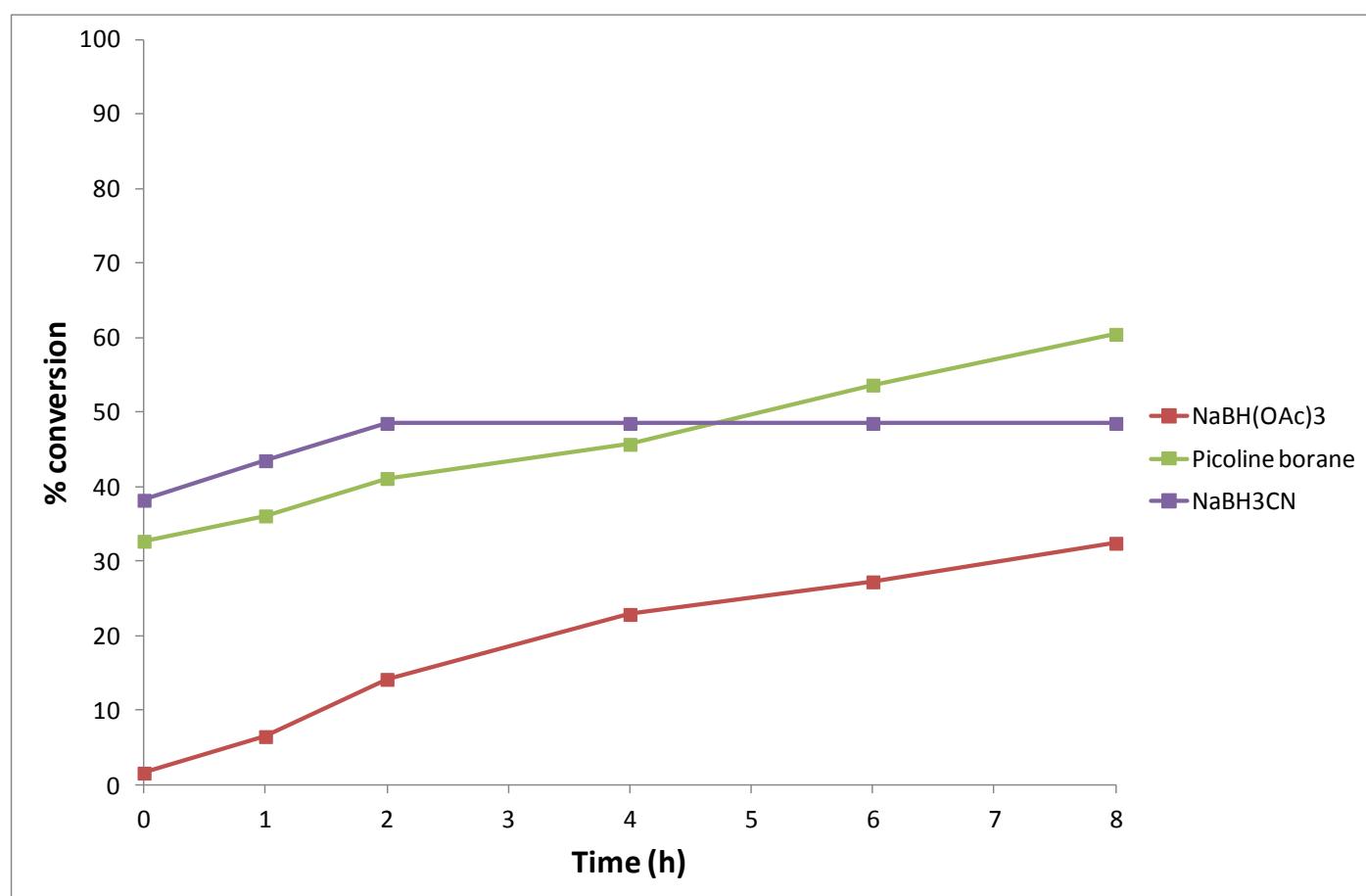
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	2.1	39.6	0.3
1	99.1	40.5	0.6
2	100.0	45.4	0.6
4	100.0	56.1	0.6
6	100.0	60.9	0.6
8	100.0	71.4	0.6
24	100.0	71.4	0.6

Reaction 1: DMC



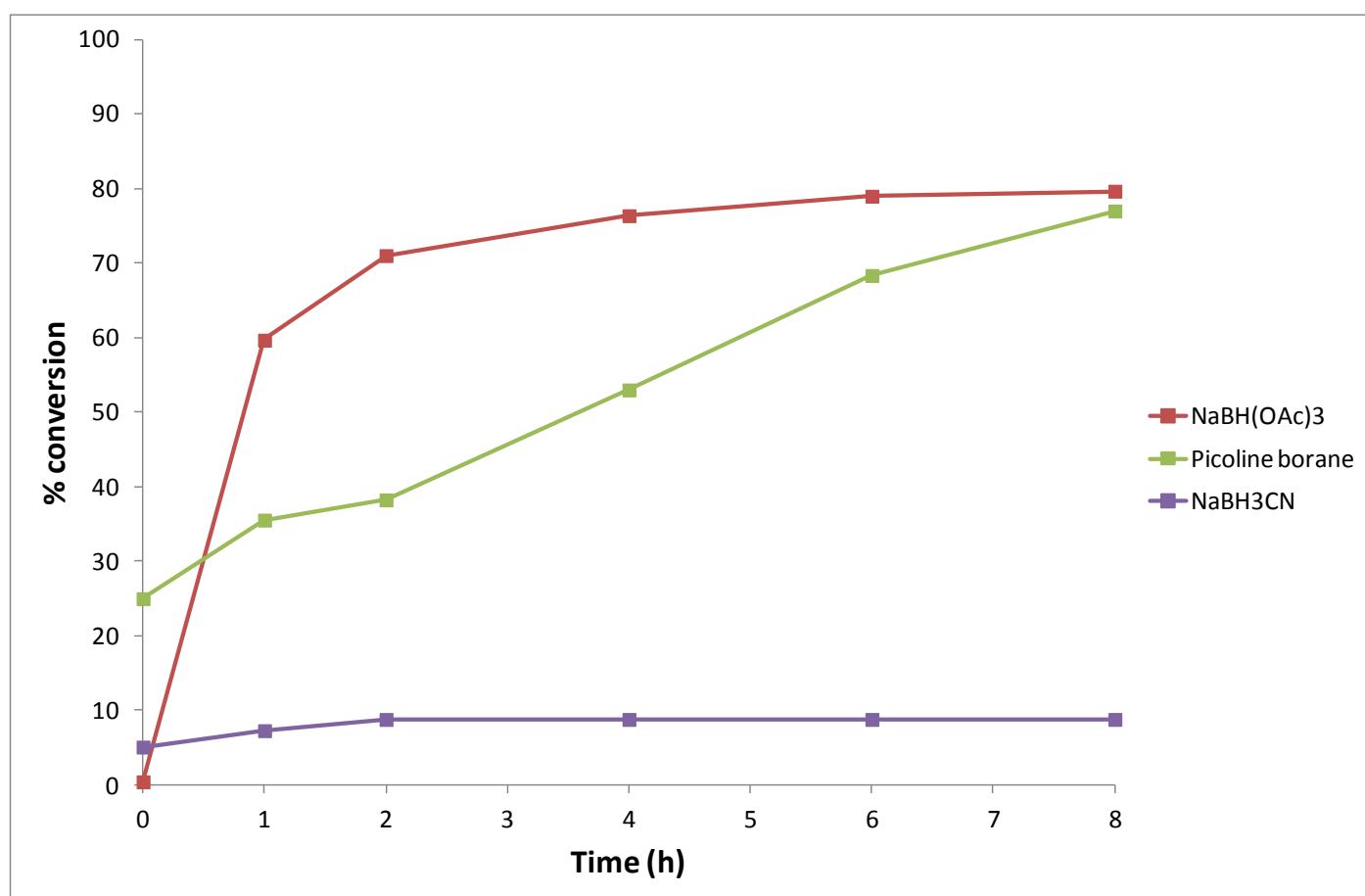
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	0.9	31.3	1.5
1	75.2	44.8	1.5
2	81.3	47.1	1.5
4	83.1	60.1	1.5
6	83.5	64.2	1.5
8	83.9	73.0	1.5
24	85.3	79.2	1.5

Reaction 1: DMF



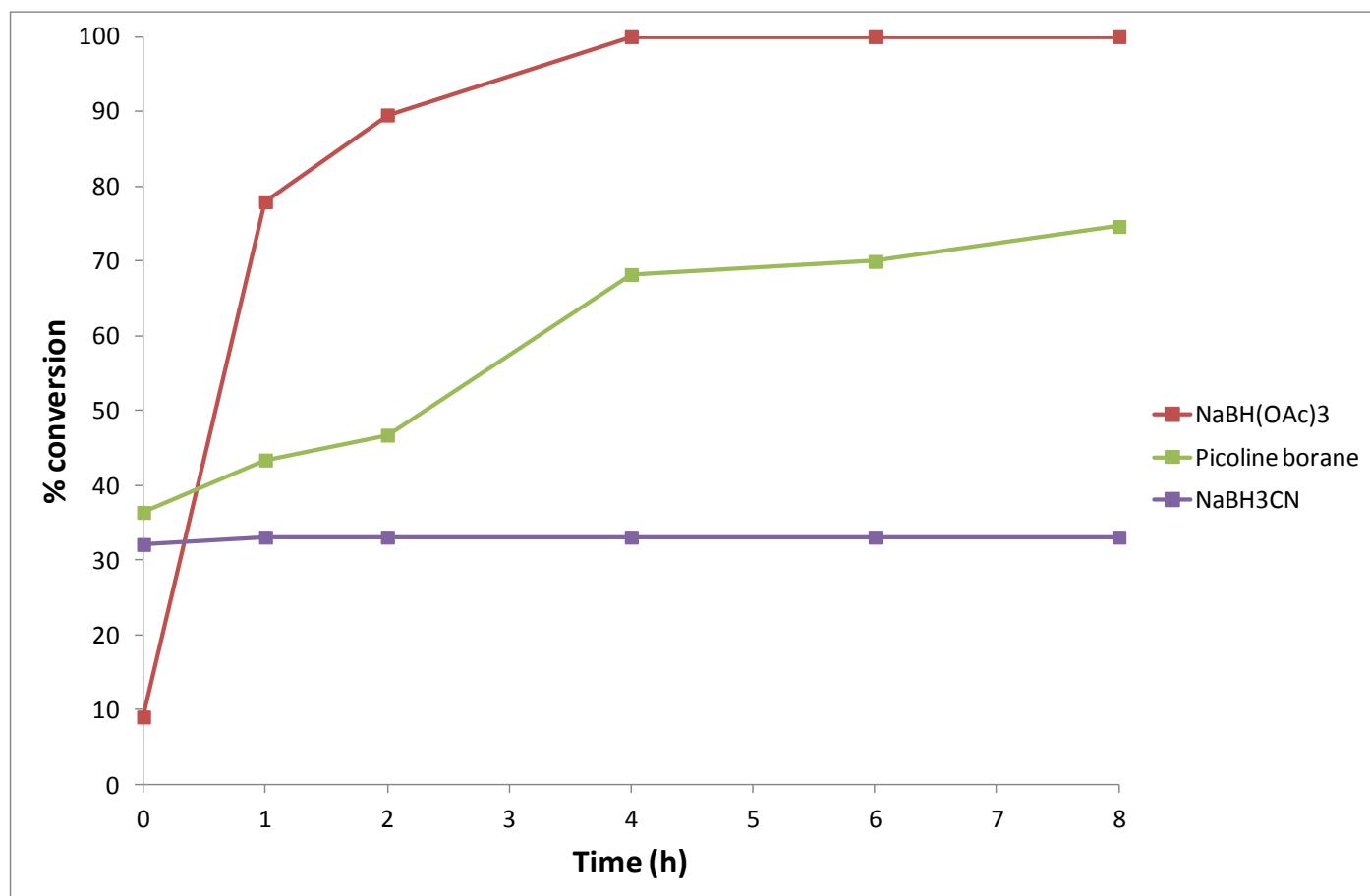
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	1.6	32.7	38.2
1	6.5	36.1	43.5
2	14.2	41.1	48.5
4	22.9	45.7	48.5
6	27.3	53.6	48.5
8	32.4	60.4	48.5
24	39.3	77.1	48.5

Reaction 1: EtOAc



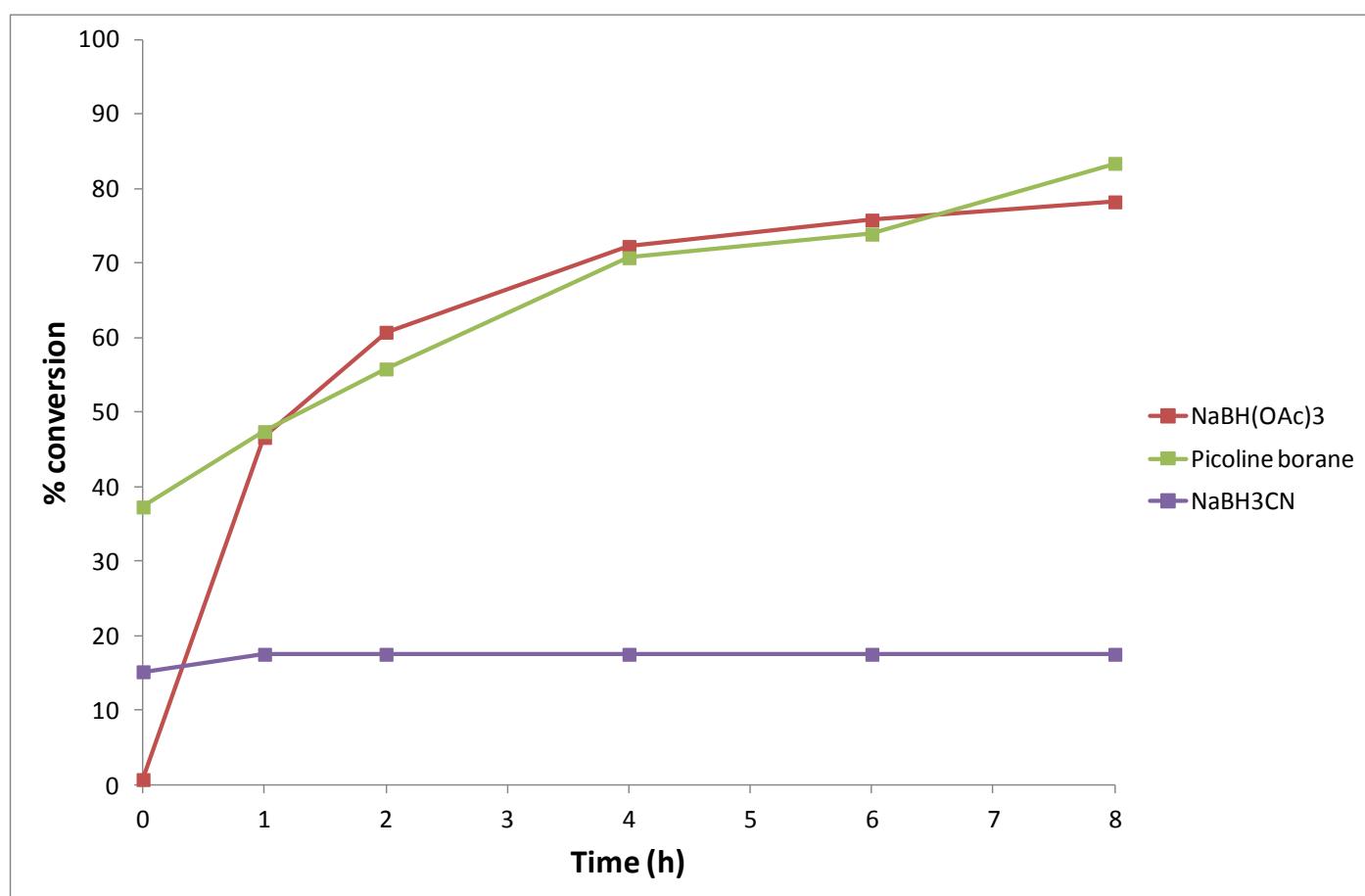
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	0.4	25.0	5.1
1	59.7	35.5	7.3
2	71.0	38.3	8.8
4	76.4	53.0	8.8
6	79.0	68.3	8.8
8	79.6	77.0	8.8
24	79.8	83.8	8.8

Reaction 1: IPA



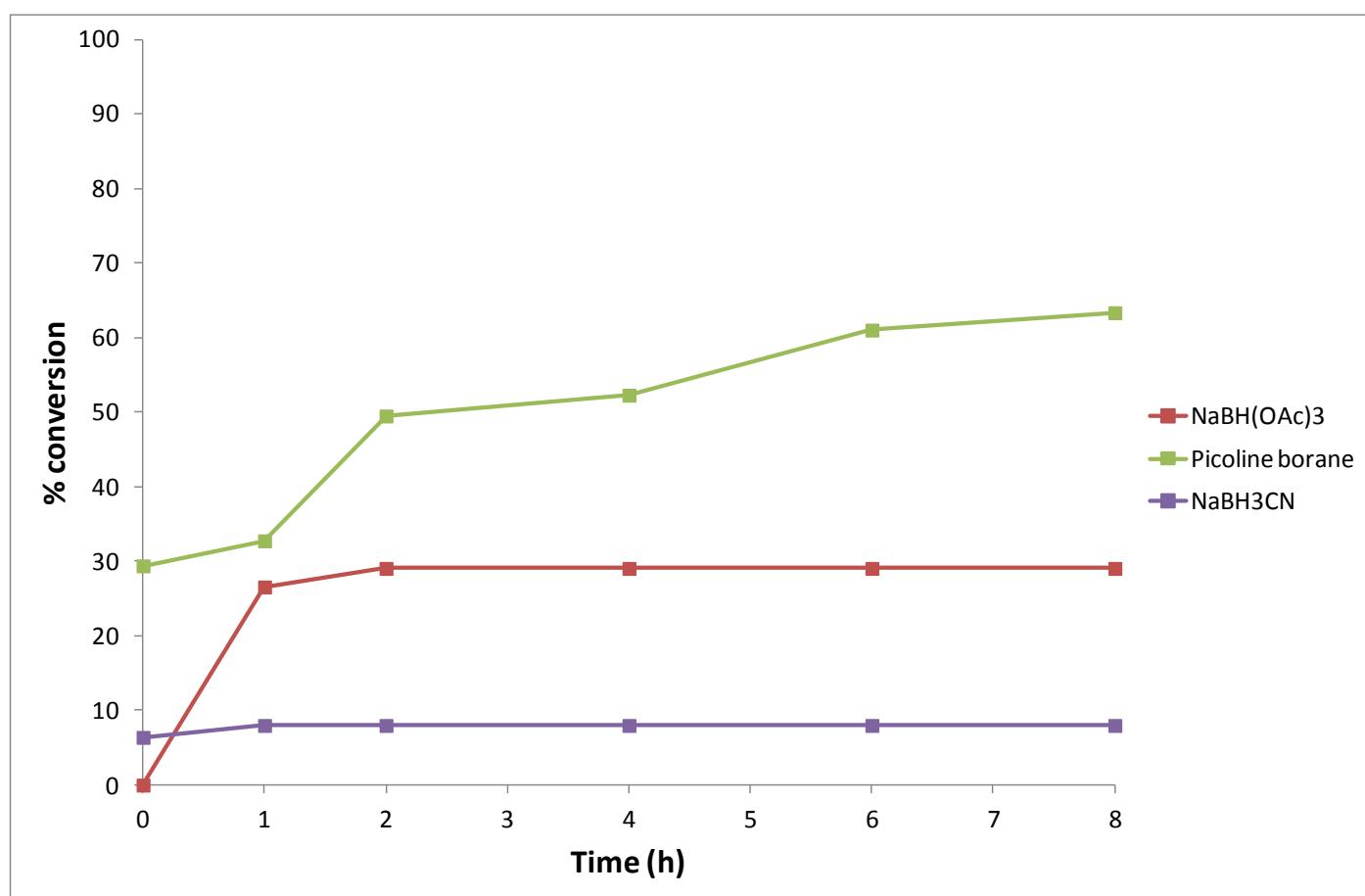
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	9.1	36.4	32.1
1	77.9	43.4	33.1
2	89.5	46.7	33.1
4	100.0	68.2	33.1
6	100.0	70.0	33.1
8	100.0	74.6	33.1
24	100.0	74.6	33.1

Reaction 1: 2-MeTHF



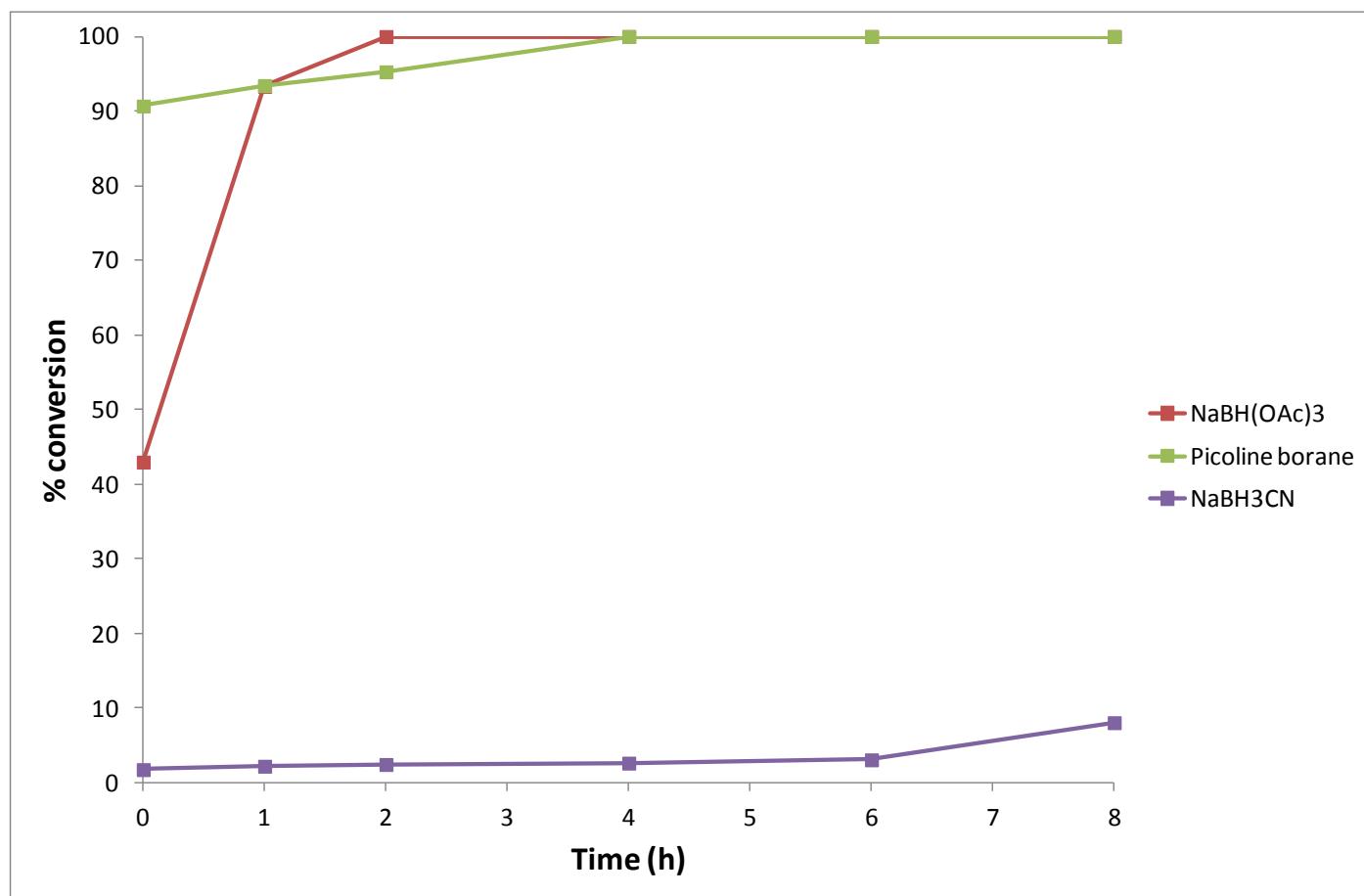
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	0.7	37.3	15.2
1	46.6	47.4	17.5
2	60.7	55.8	17.5
4	72.2	70.7	17.5
6	75.8	73.9	17.5
8	78.2	83.4	17.5
24	79.6	83.4	17.5

Reaction 1: THF



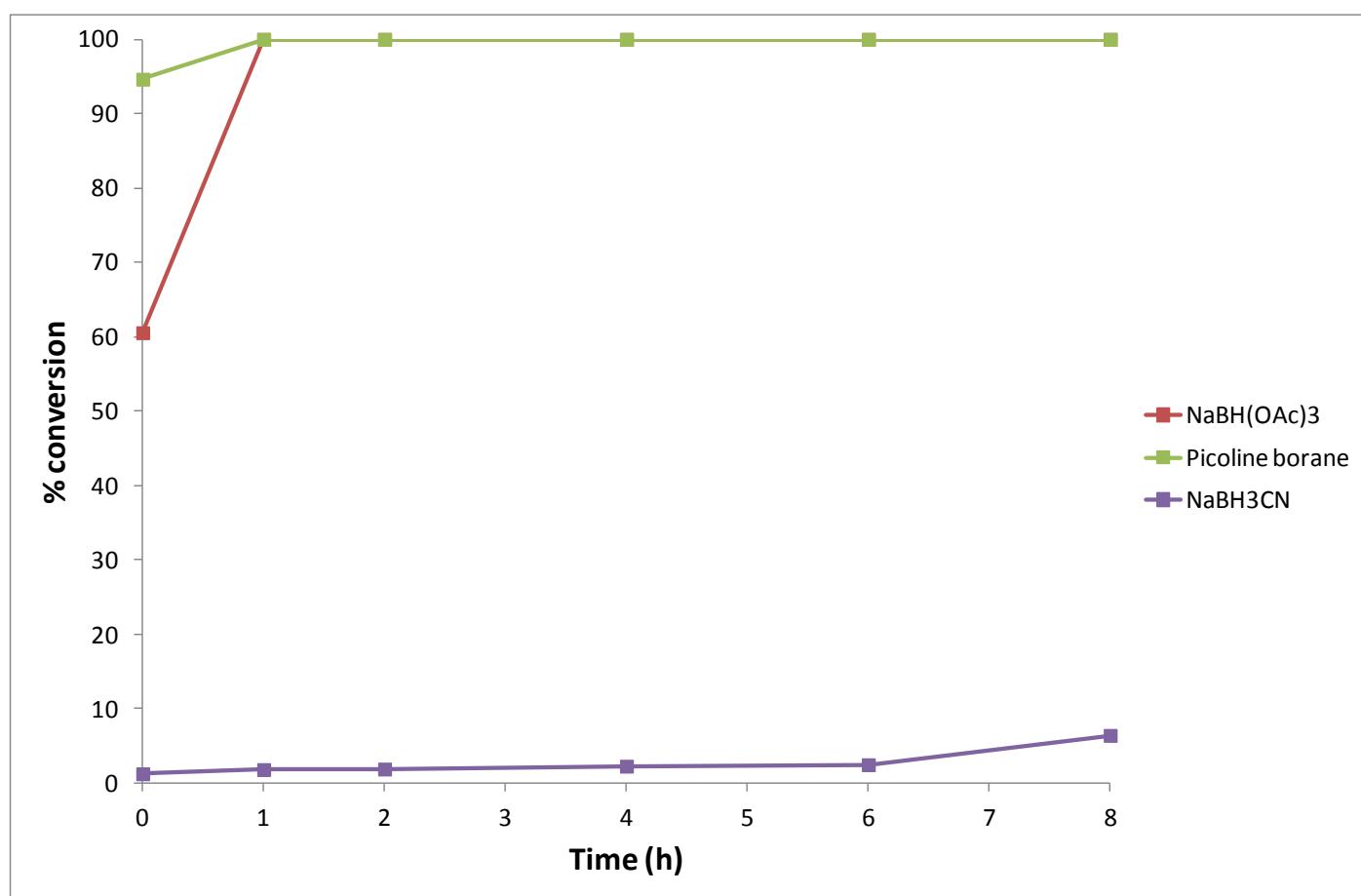
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	0.0	29.4	6.4
1	26.6	32.8	8.0
2	29.1	49.5	8.0
4	29.1	52.3	8.0
6	29.1	61.0	8.0
8	29.1	63.4	8.0
24	29.1	63.4	8.0

Reaction 2: TBME



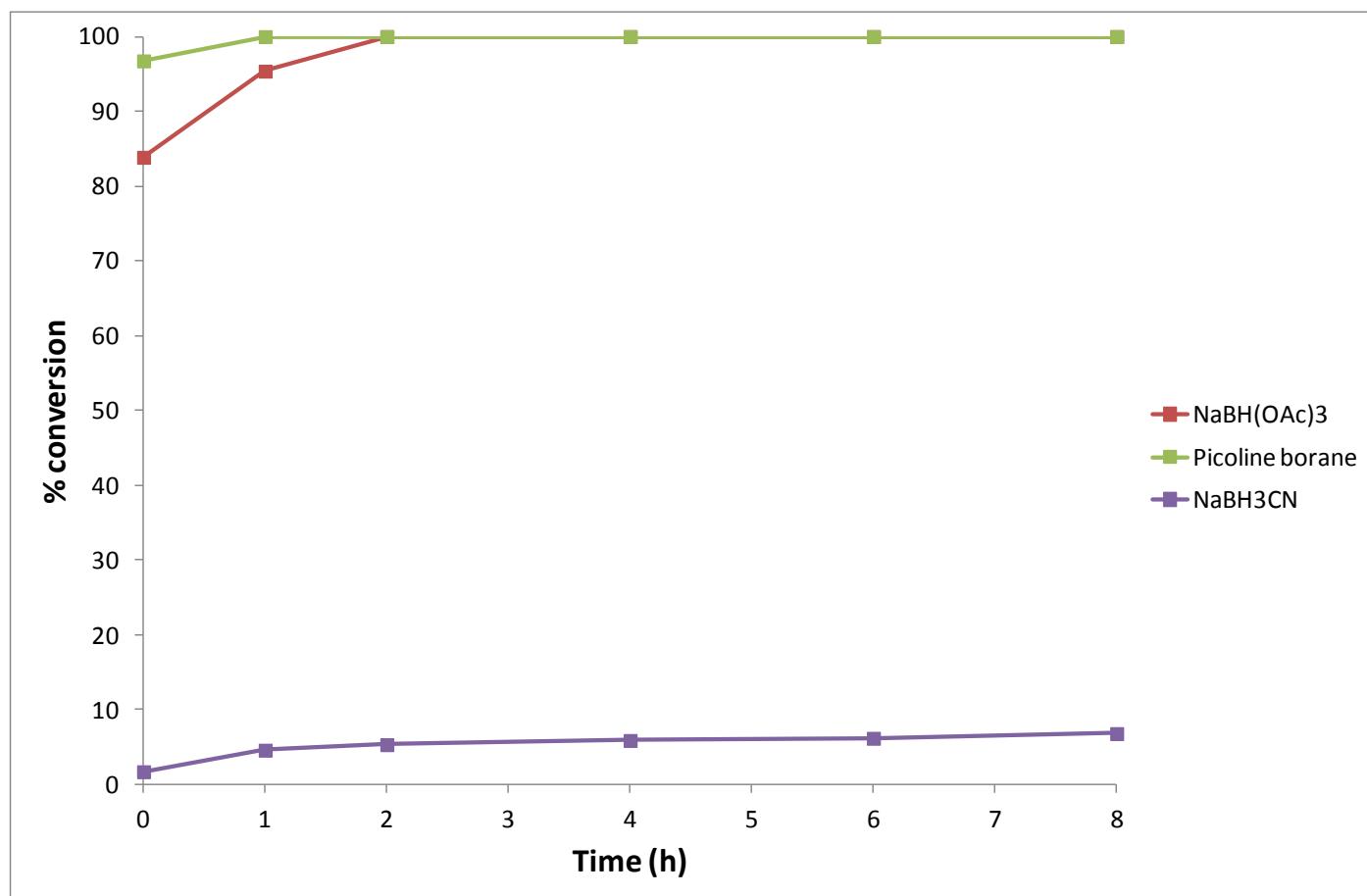
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	43.0	90.7	1.8
1	93.3	93.4	2.2
2	100.0	95.3	2.5
4	100.0	100.0	2.7
6	100.0	100.0	3.1
8	100.0	100.0	8.1
24	100.0	100.0	12.6

Reaction 2: CPME



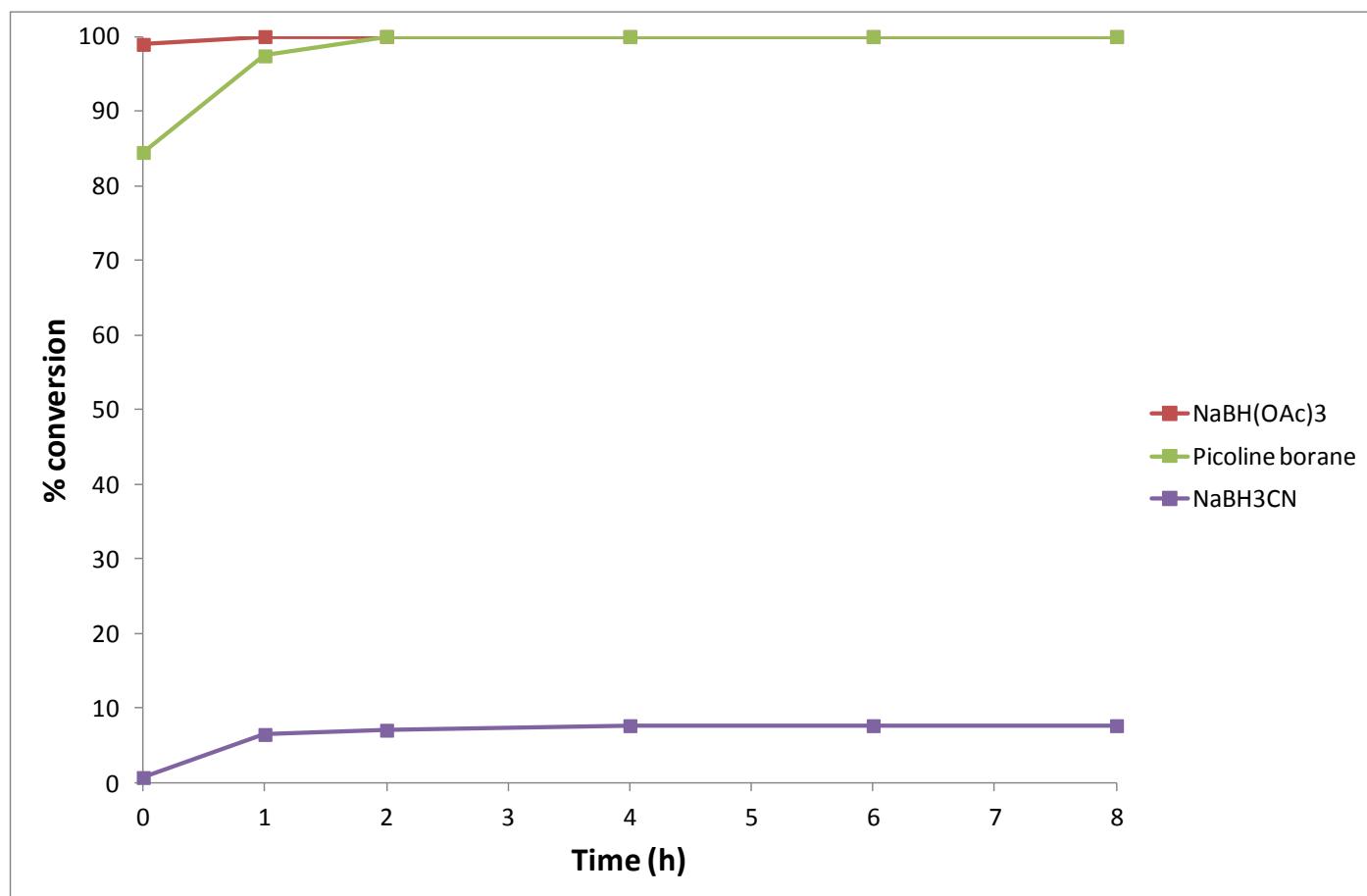
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	60.6	94.6	1.3
1	100.0	100.0	1.8
2	100.0	100.0	1.9
4	100.0	100.0	2.3
6	100.0	100.0	2.5
8	100.0	100.0	6.4
24	100.0	100.0	8.5

Reaction 2: DCE



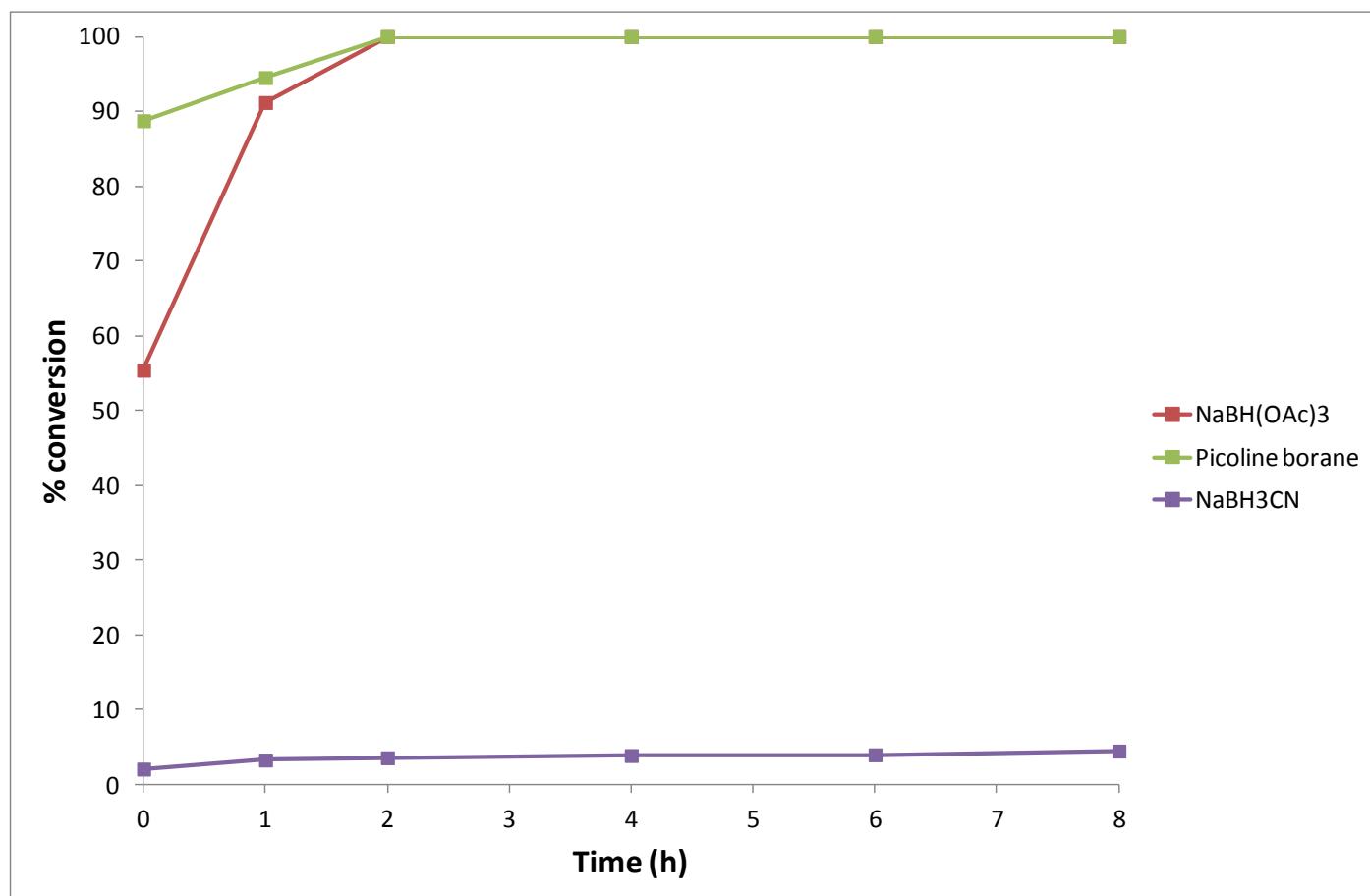
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	83.9	96.8	1.7
1	95.4	100.0	4.6
2	100.0	100.0	5.3
4	100.0	100.0	5.9
6	100.0	100.0	6.2
8	100.0	100.0	6.8
24	100.0	100.0	6.8

Reaction 2: CH₂Cl₂



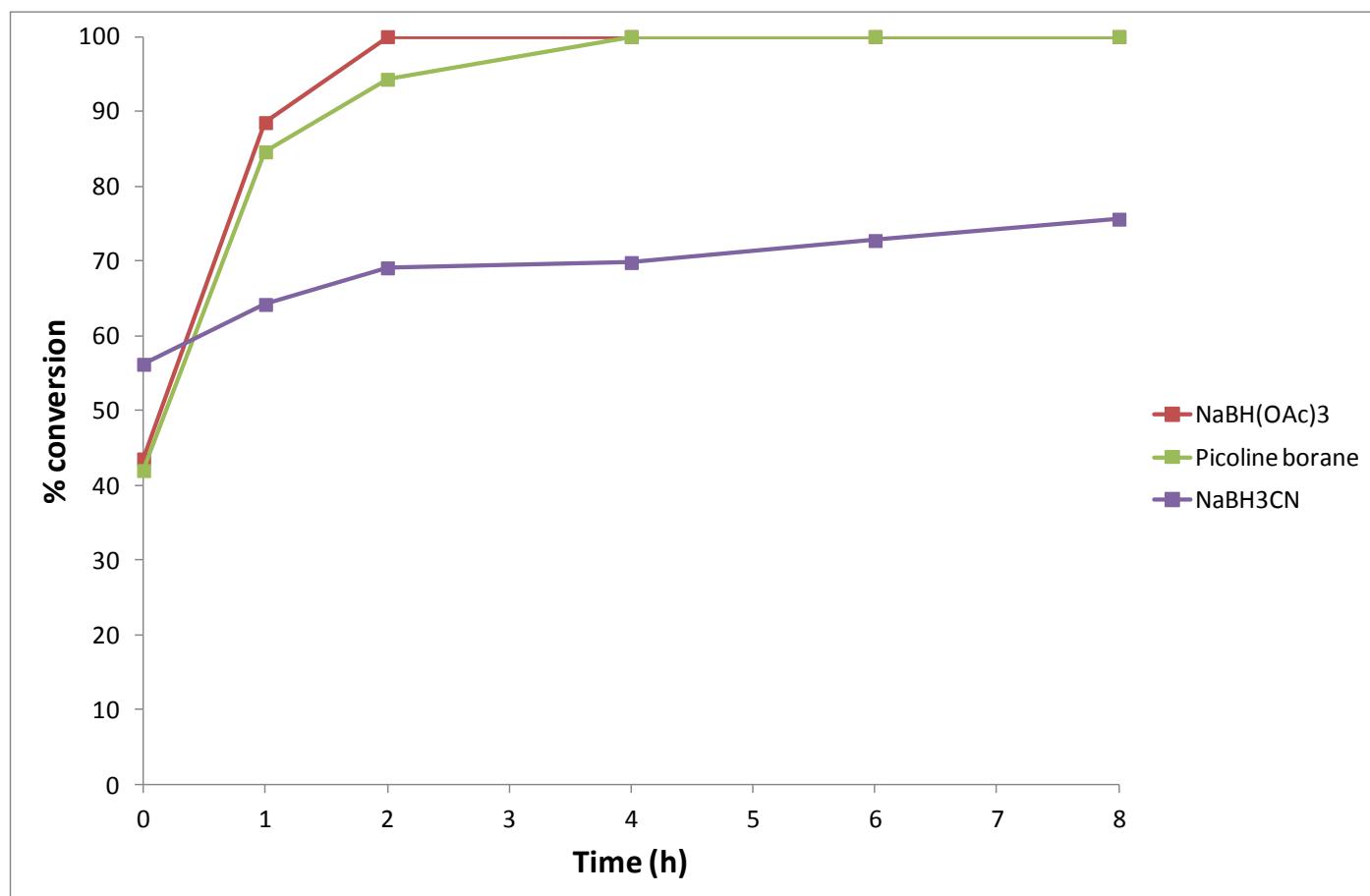
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	98.9	84.5	0.7
1	100.0	97.4	6.5
2	100.0	100.0	7.1
4	100.0	100.0	7.7
6	100.0	100.0	7.7
8	100.0	100.0	7.7
24	100.0	100.0	7.7

Reaction 2: DMC



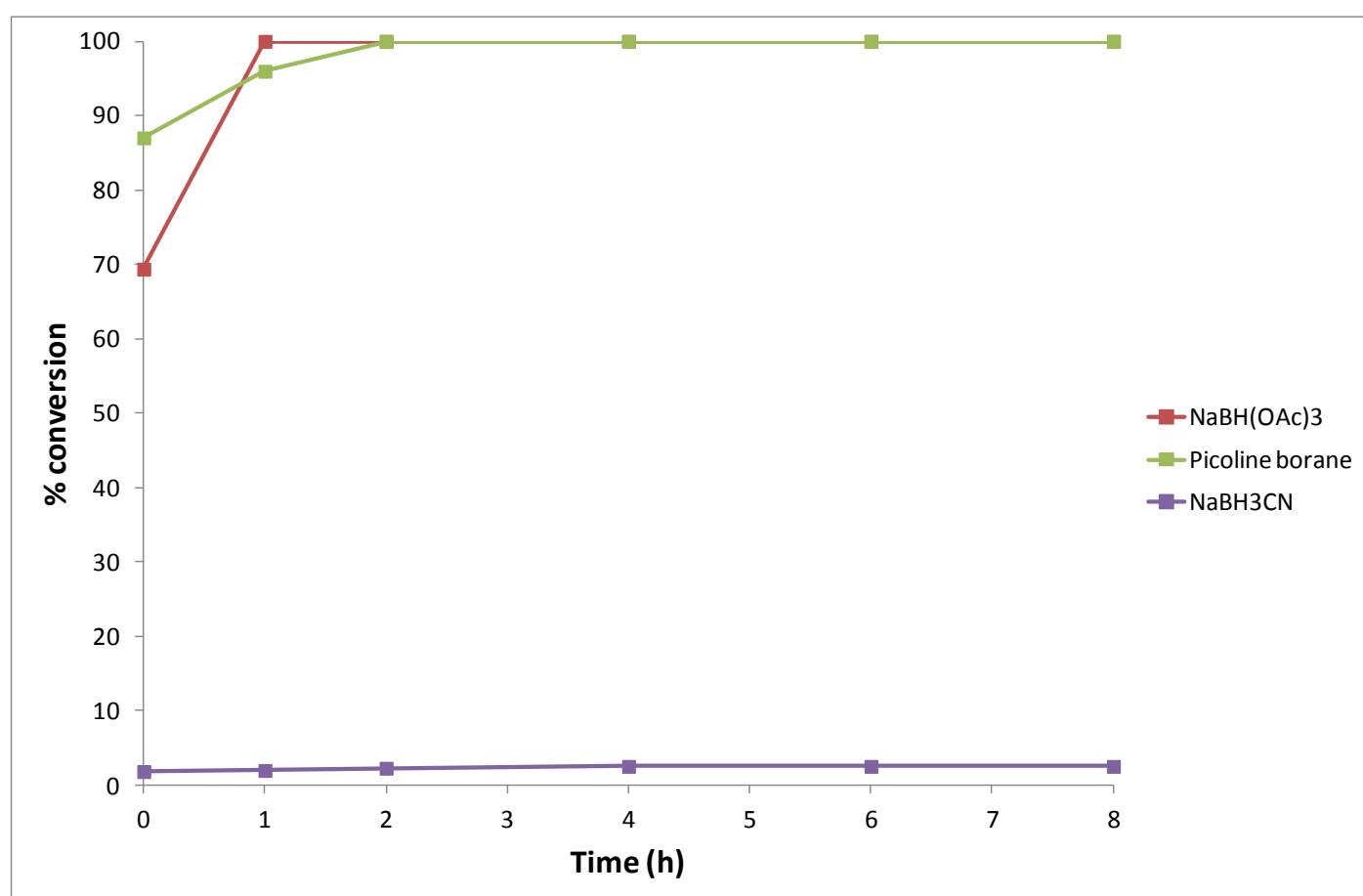
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	55.4	88.8	2.1
1	91.2	94.5	3.3
2	100.0	100.0	3.6
4	100.0	100.0	3.9
6	100.0	100.0	4.0
8	100.0	100.0	4.5
24	100.0	100.0	5.5

Reaction 2: DMF



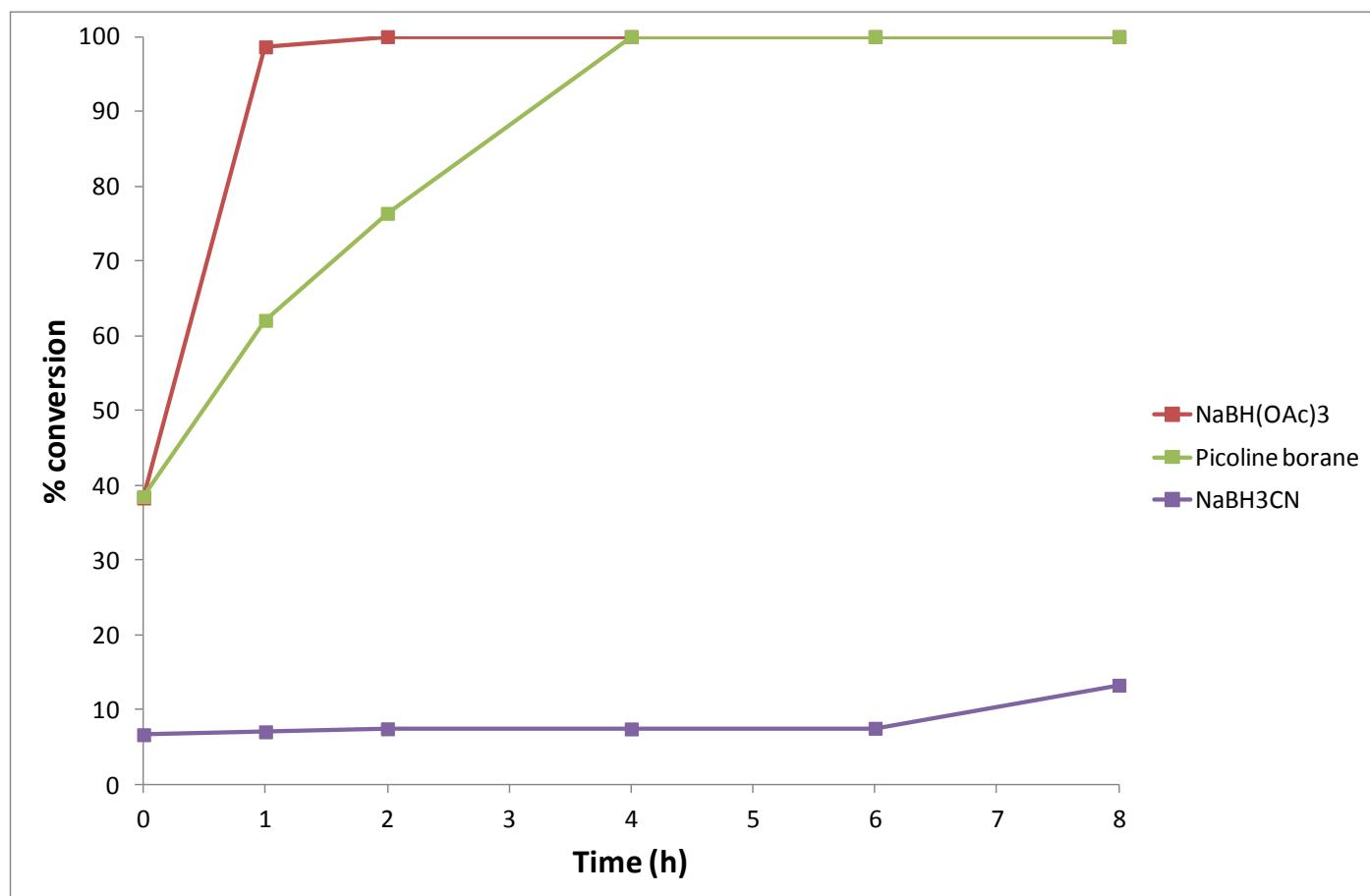
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	43.5	42.0	56.2
1	88.5	84.6	64.2
2	100.0	94.3	69.1
4	100.0	100.0	69.8
6	100.0	100.0	72.7
8	100.0	100.0	75.6
24	100.0	100.0	100.0

Reaction 2: EtOAc



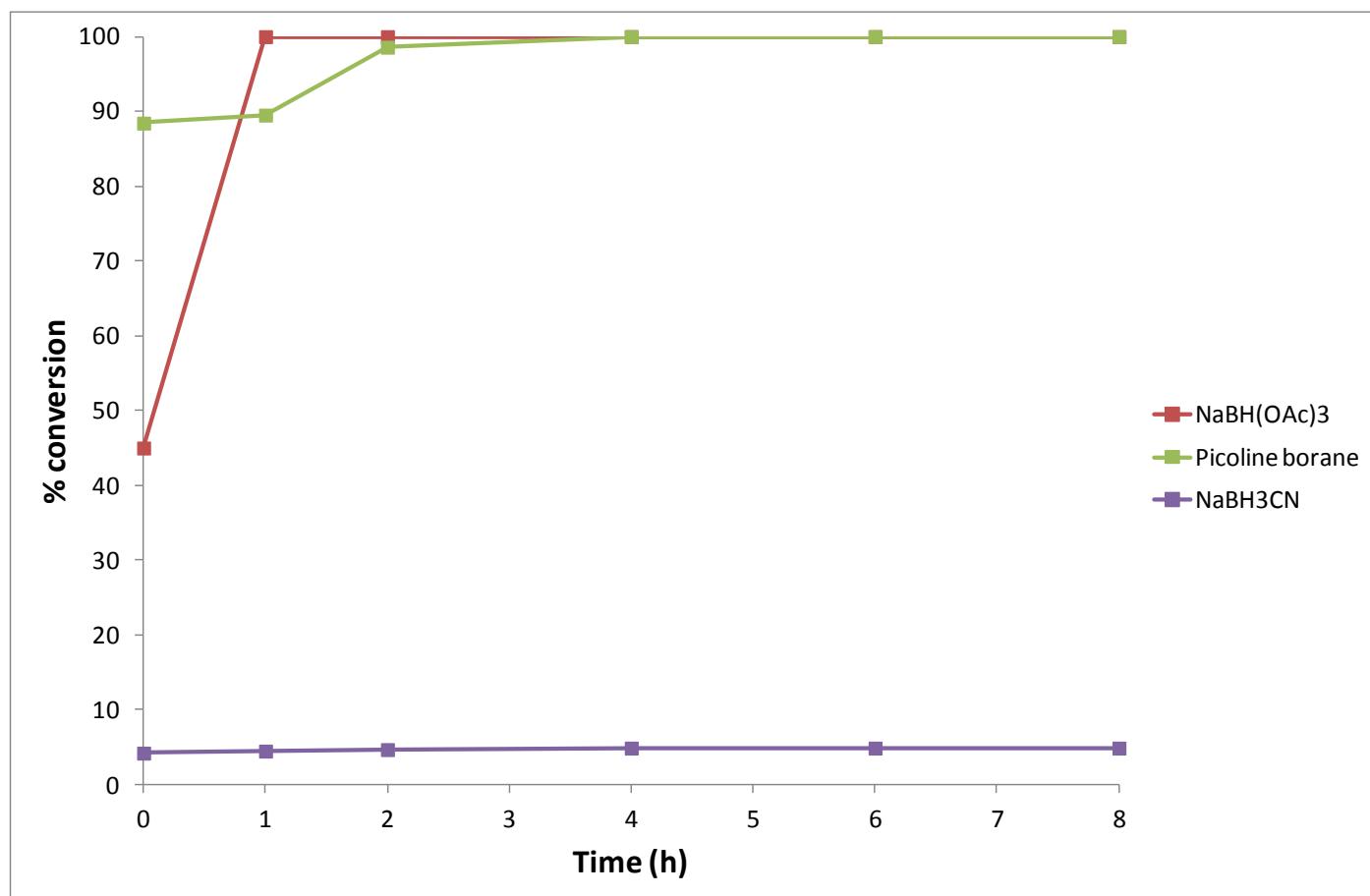
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	69.4	87.0	1.9
1	100.0	96.0	2.0
2	100.0	100.0	2.3
4	100.0	100.0	2.5
6	100.0	100.0	2.5
8	100.0	100.0	2.5
24	100.0	100.0	2.5

Reaction 2: IPA



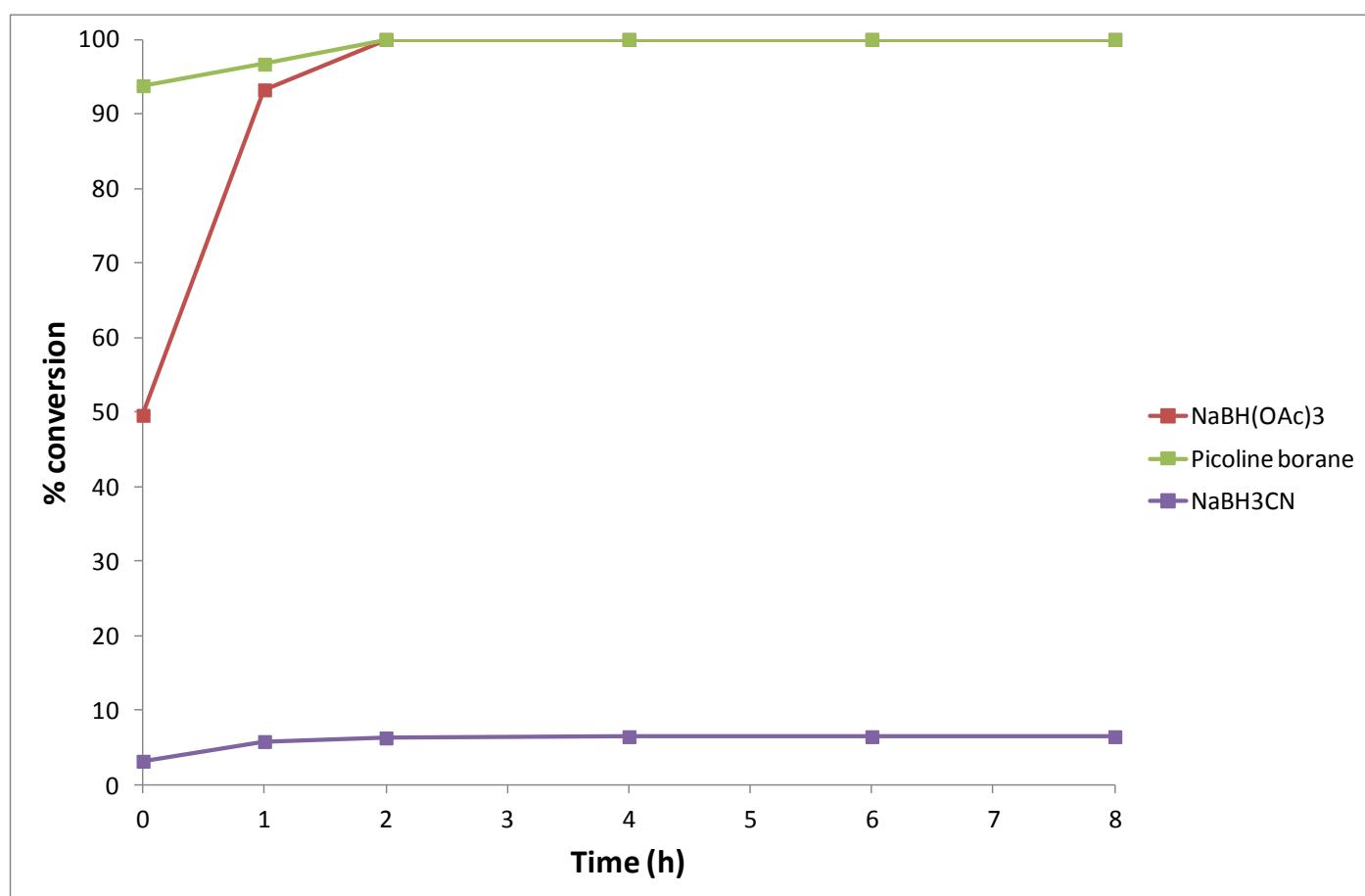
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	38.3	38.5	6.7
1	98.6	62.1	7.1
2	100.0	76.4	7.5
4	100.0	100.0	7.5
6	100.0	100.0	7.5
8	100.0	100.0	13.3
24	100.0	100.0	13.3

Reaction 2: 2-MeTHF



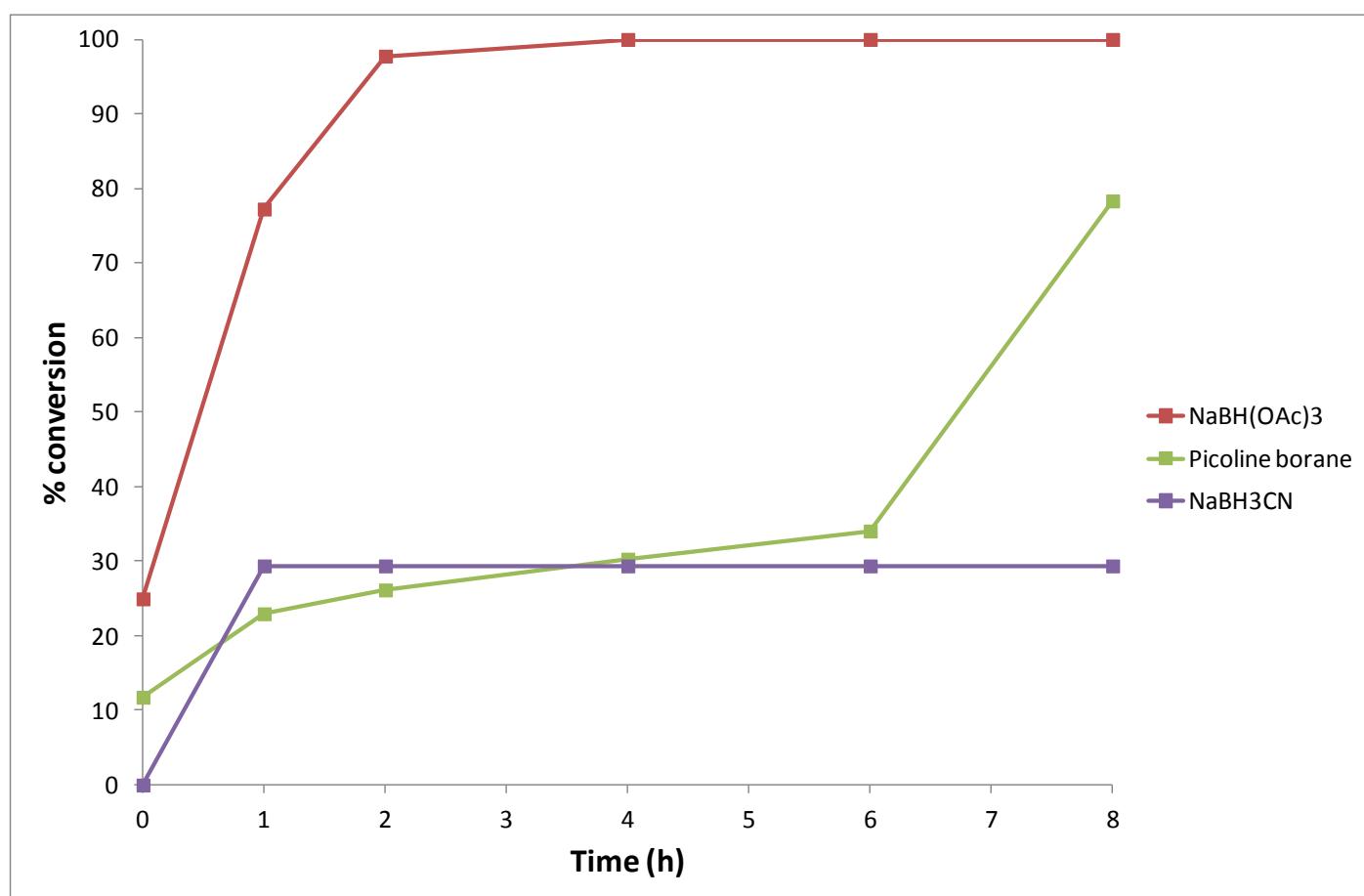
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	45.0	88.4	4.2
1	100.0	89.5	4.5
2	100.0	98.6	4.7
4	100.0	100.0	4.9
6	100.0	100.0	4.9
8	100.0	100.0	4.9
24	100.0	100.0	4.9

Reaction 2: THF



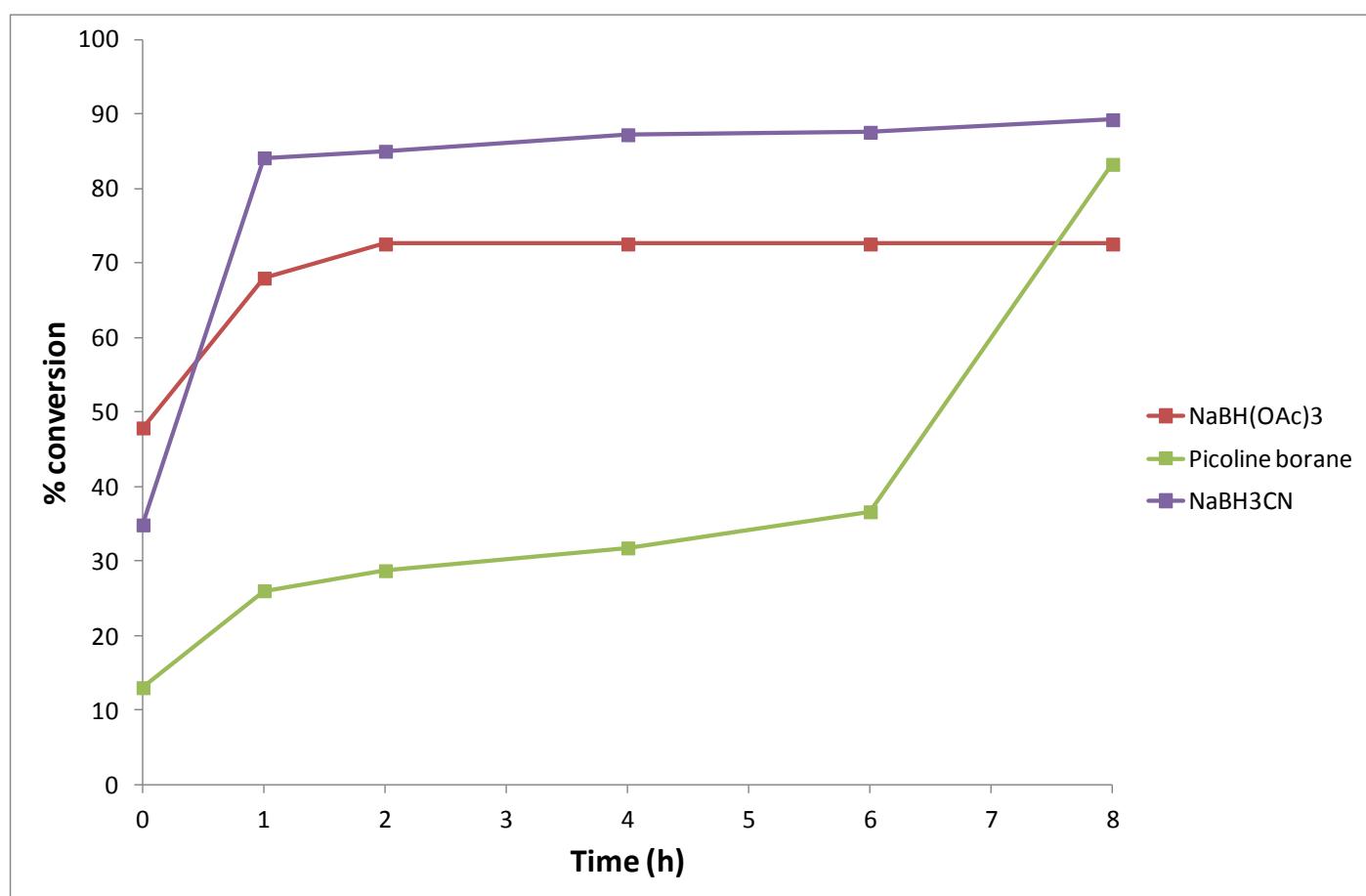
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	49.6	93.8	3.2
1	93.2	96.7	5.8
2	100.0	100.0	6.3
4	100.0	100.0	6.5
6	100.0	100.0	6.5
8	100.0	100.0	6.5
24	100.0	100.0	6.5

Reaction 3: TBME



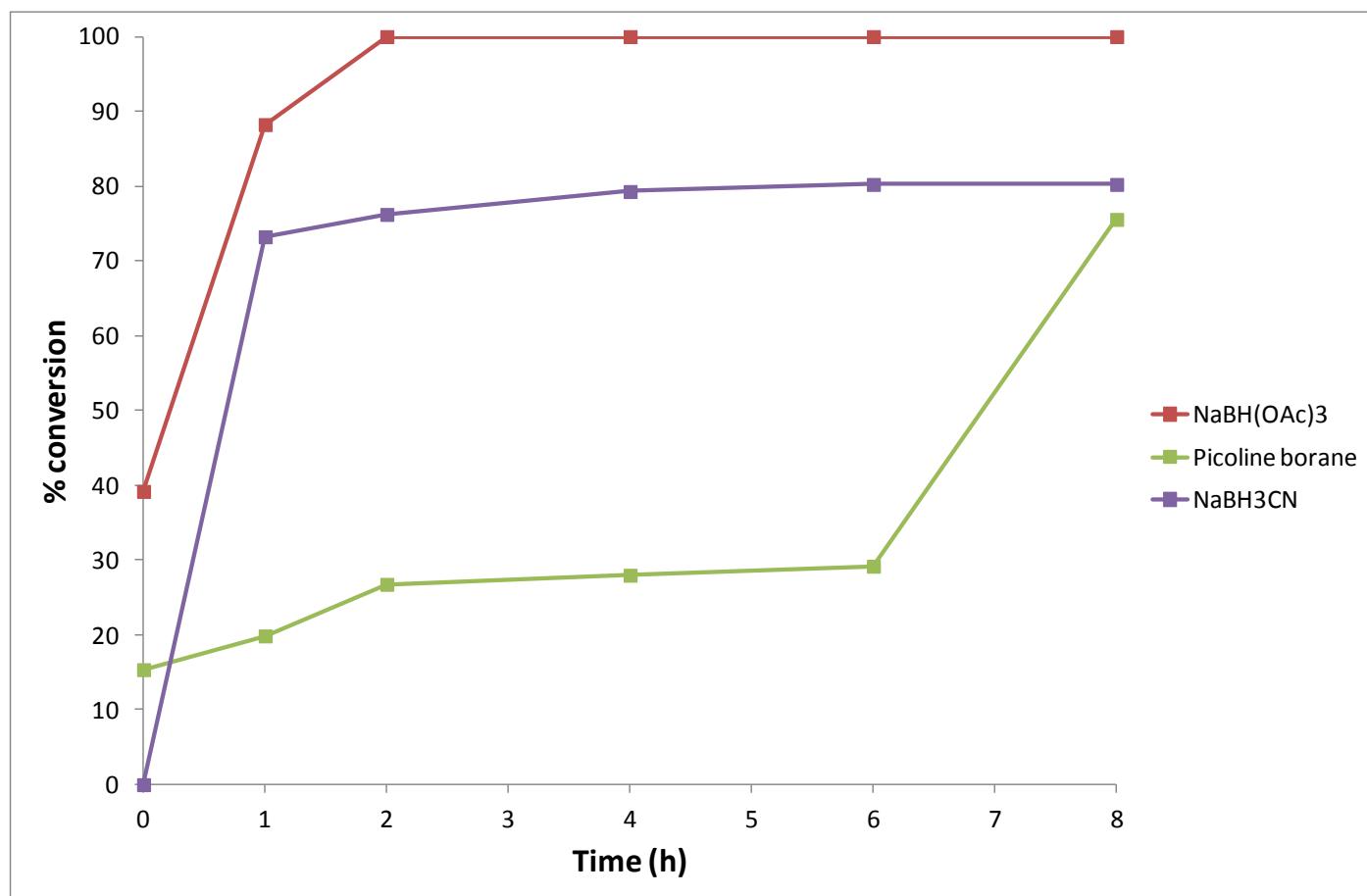
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	25.0	11.8	0.0
1	77.3	23.0	29.4
2	97.8	26.2	29.4
4	100.0	30.3	29.4
6	100.0	34.1	29.4
8	100.0	78.3	29.4
24	100.0	78.3	29.4

Reaction 3: CPME



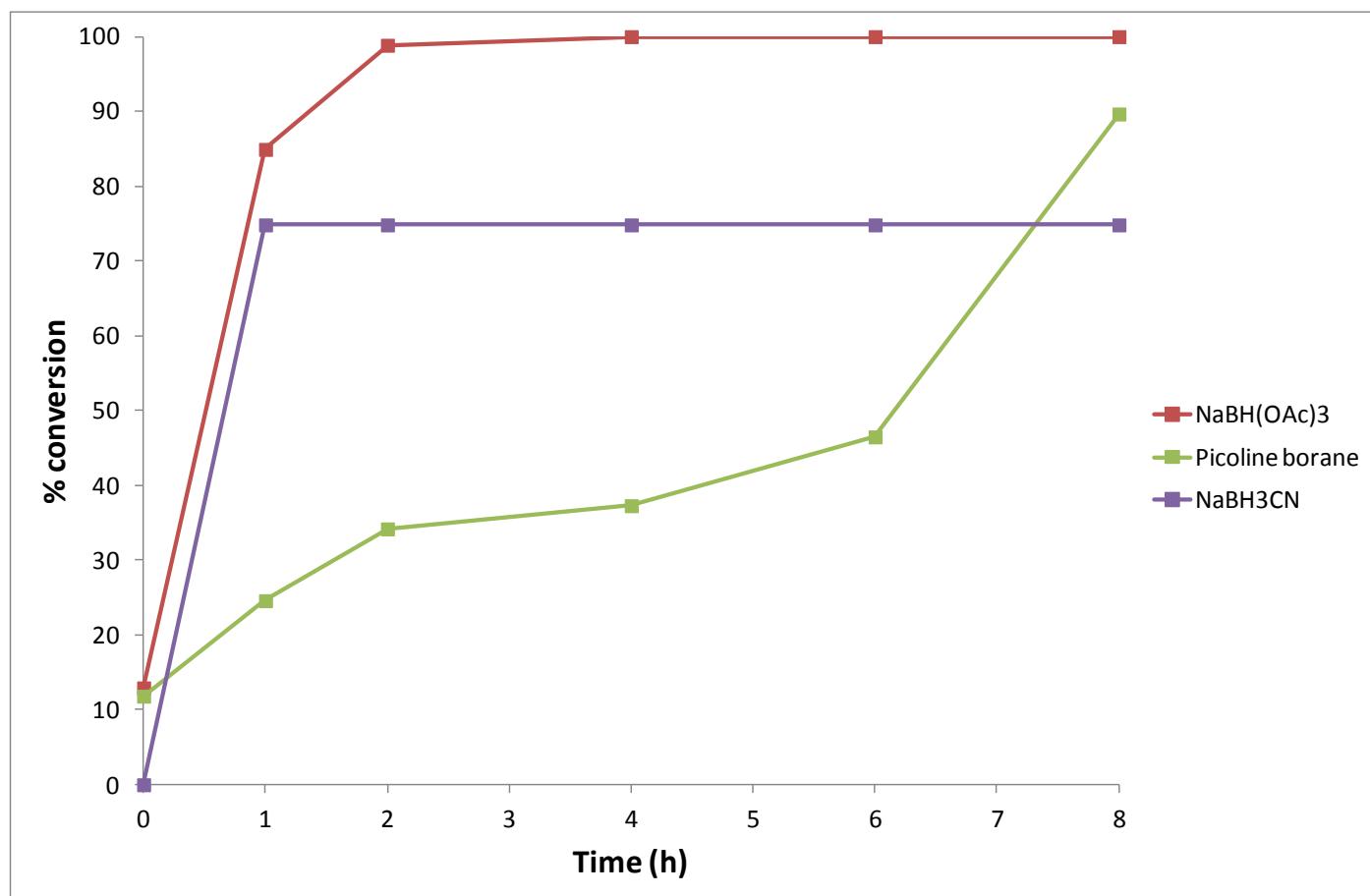
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	47.9	13.1	34.9
1	68.0	26.1	84.1
2	72.6	28.7	85.0
4	72.6	31.8	87.2
6	72.6	36.6	87.5
8	72.6	83.2	89.2
24	72.6	83.2	100.0

Reaction 3: DCE



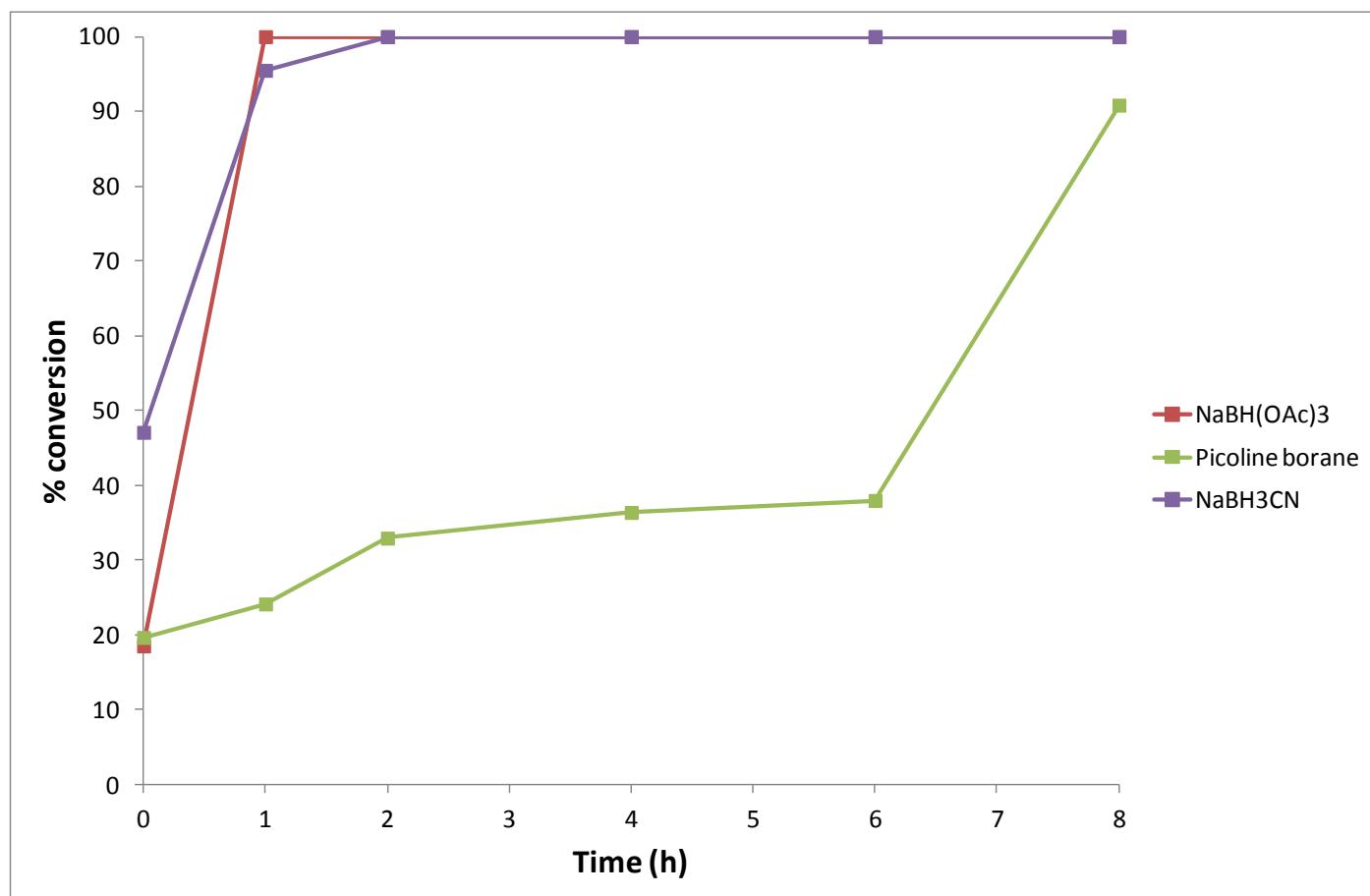
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	39.2	15.4	0.0
1	88.2	19.9	73.2
2	100.0	26.8	76.2
4	100.0	28.0	79.3
6	100.0	29.2	80.2
8	100.0	75.6	80.2
24	100.0	75.6	80.2

Reaction 3: CH₂Cl₂



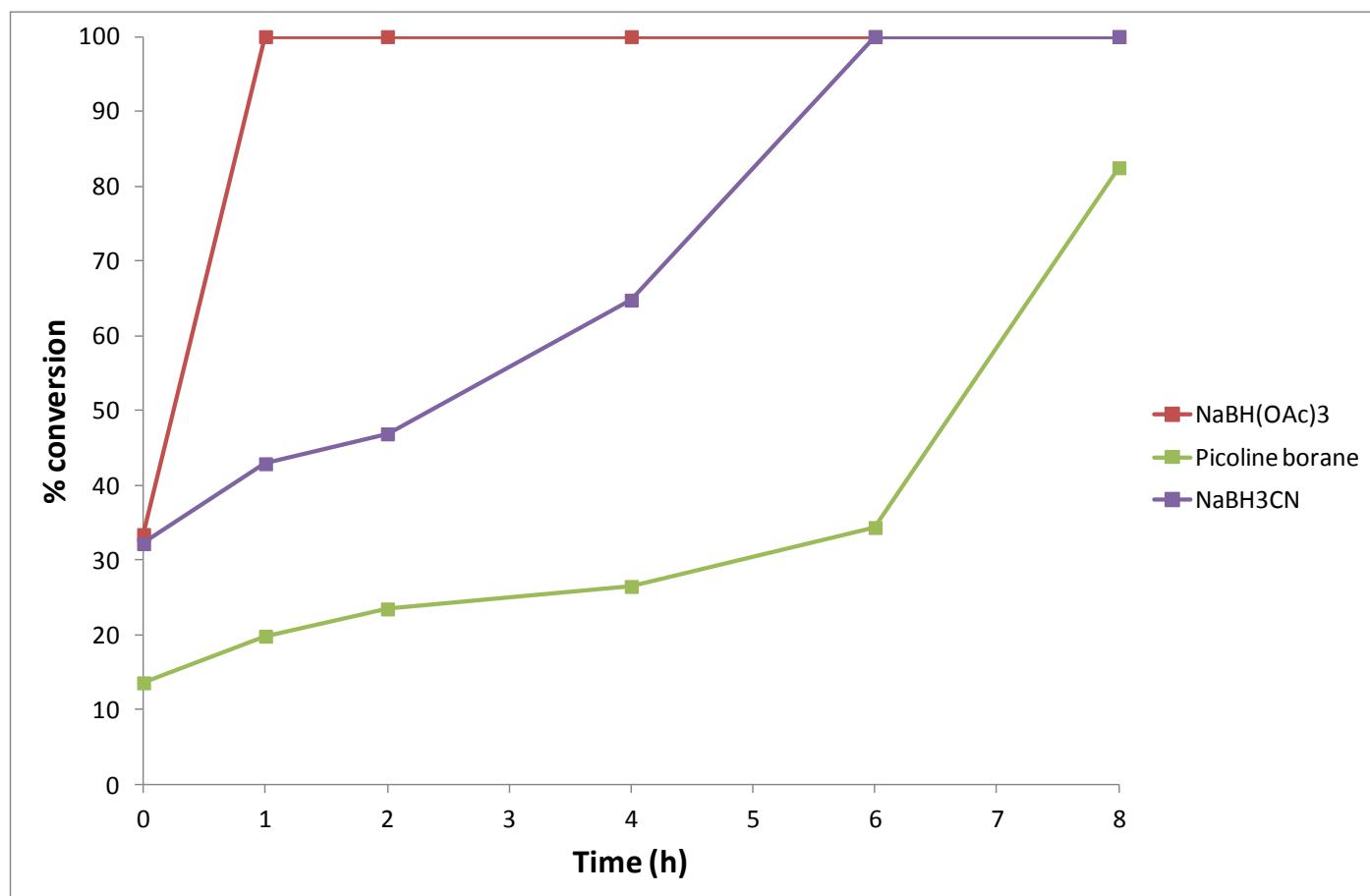
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	12.9	11.8	0.0
1	84.9	24.6	74.8
2	98.8	34.2	74.8
4	100.0	37.3	74.8
6	100.0	46.5	74.8
8	100.0	89.7	74.8
24	100.0	89.7	74.8

Reaction 3: DMC



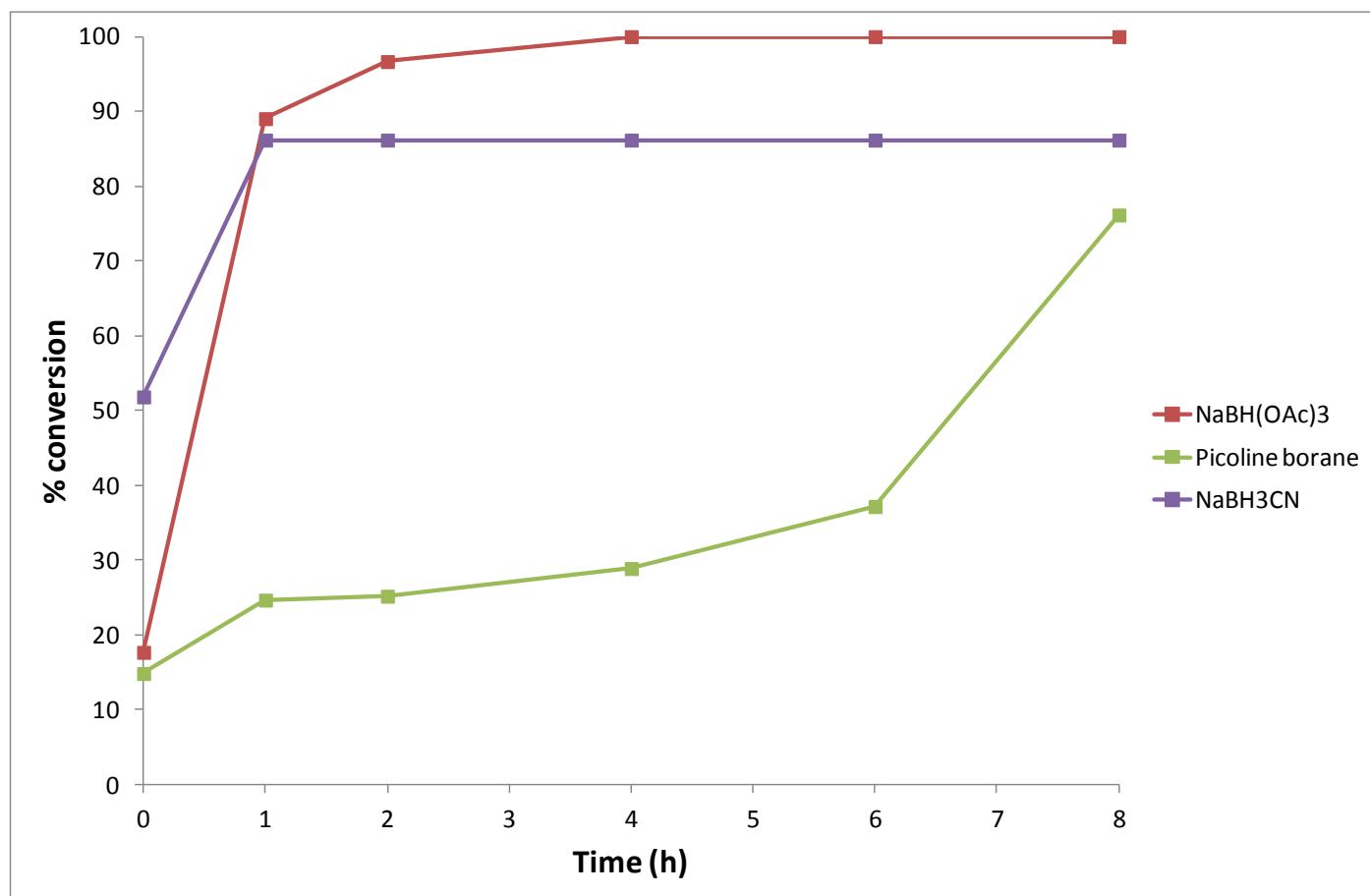
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	18.6	19.7	47.1
1	100.0	24.2	95.5
2	100.0	33.0	100.0
4	100.0	36.4	100.0
6	100.0	38.0	100.0
8	100.0	90.8	100.0
24	100.0	93.2	100.0

Reaction 3: DMF



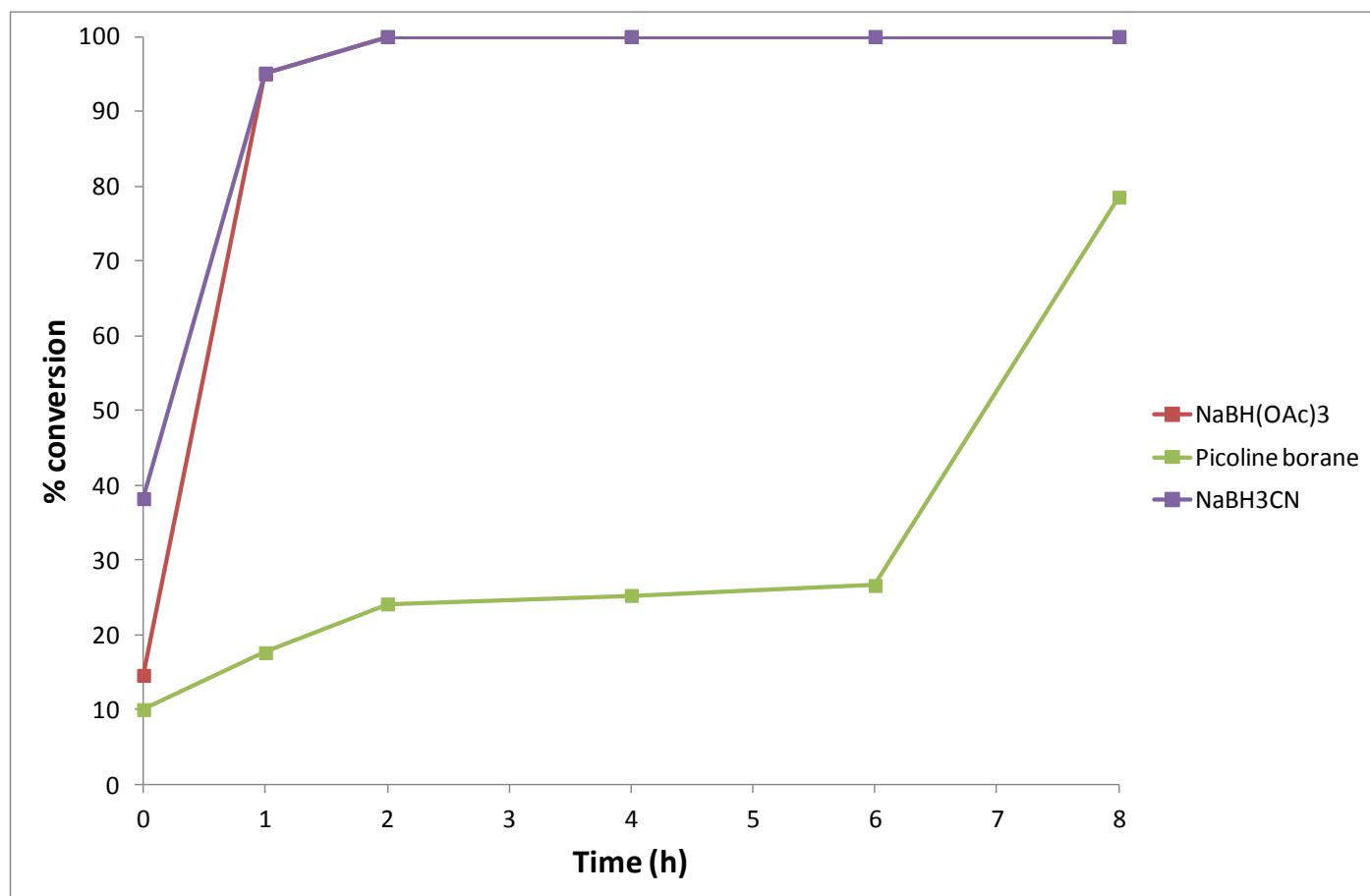
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	33.4	13.6	32.2
1	100.0	19.9	42.9
2	100.0	23.5	46.9
4	100.0	26.5	64.8
6	100.0	34.4	100.0
8	100.0	82.5	100.0
24	100.0	83.0	100.0

Reaction 3: EtOAc



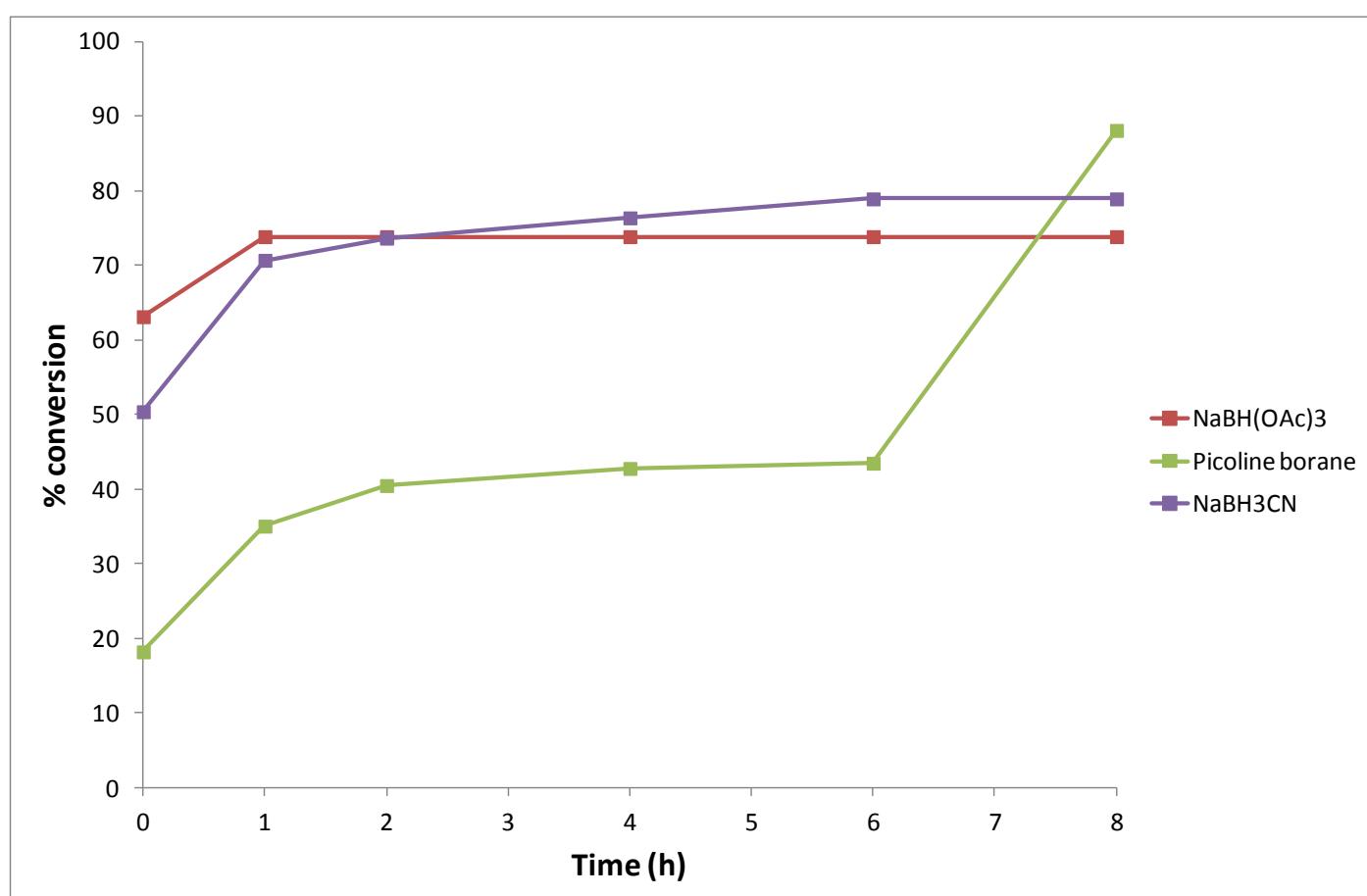
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	17.7	14.9	51.8
1	89.0	24.7	86.2
2	96.7	25.2	86.2
4	100.0	28.9	86.2
6	100.0	37.2	86.2
8	100.0	76.2	86.2
24	100.0	76.2	86.2

Reaction 3: IPA



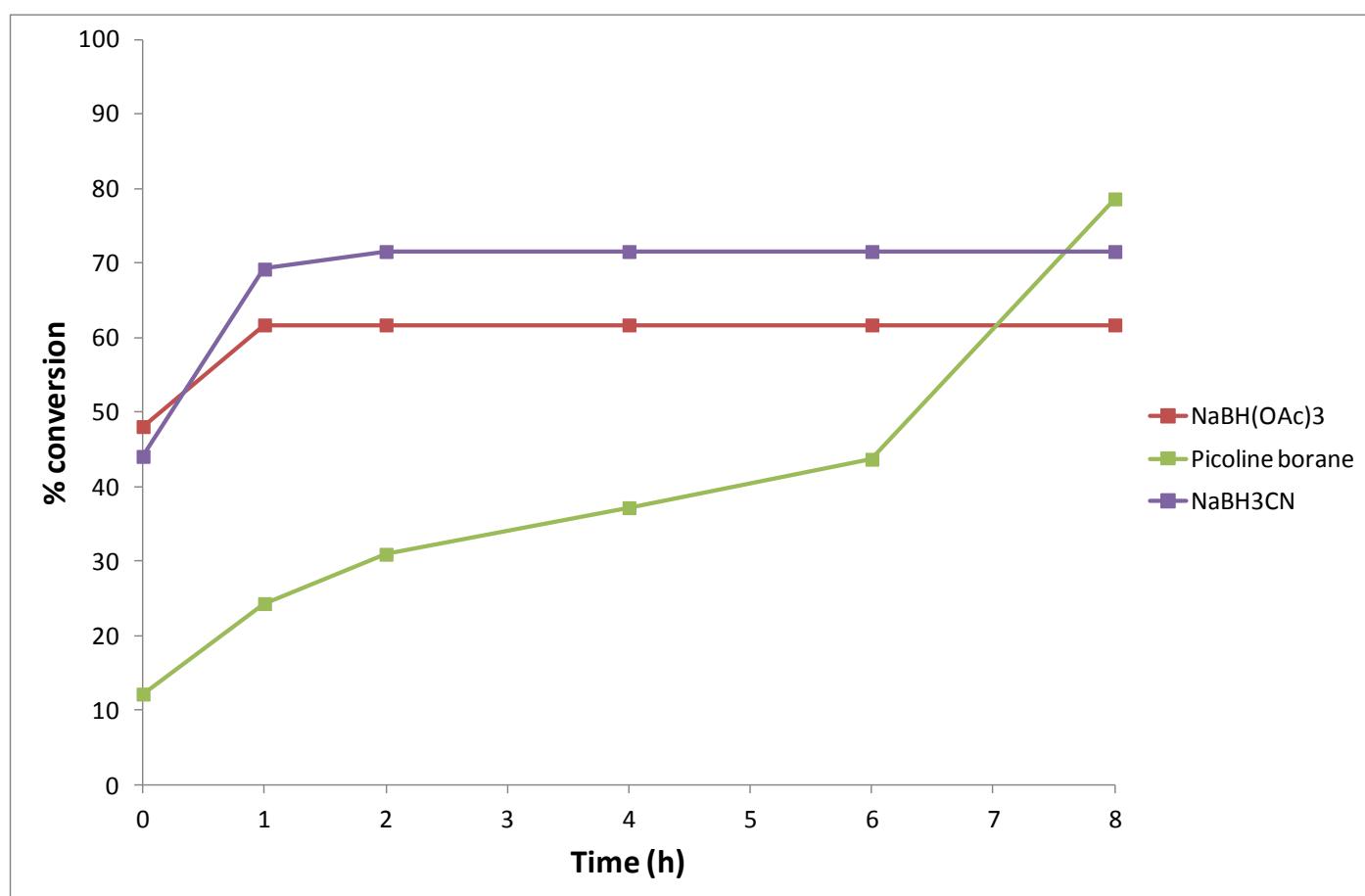
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	14.6	10.0	38.2
1	95.0	17.7	95.1
2	100.0	24.2	100.0
4	100.0	25.3	100.0
6	100.0	26.6	100.0
8	100.0	78.5	100.0
24	100.0	78.5	100.0

Reaction 3: 2-MeTHF



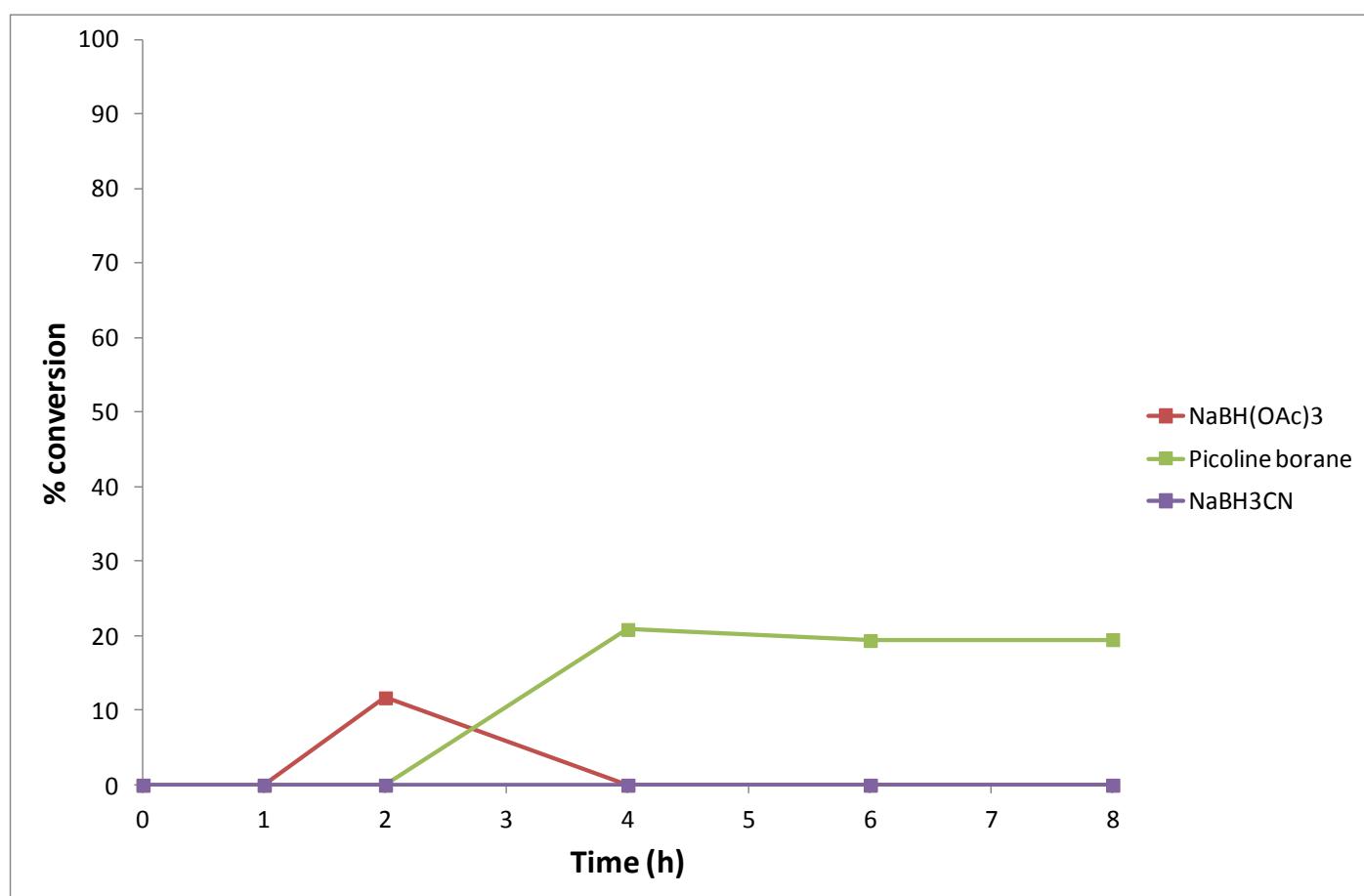
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	63.1	18.2	50.4
1	73.9	35.1	70.7
2	73.9	40.5	73.7
4	73.9	42.8	76.4
6	73.9	43.5	78.9
8	73.9	88.1	78.9
24	73.9	88.1	78.9

Reaction 3: THF



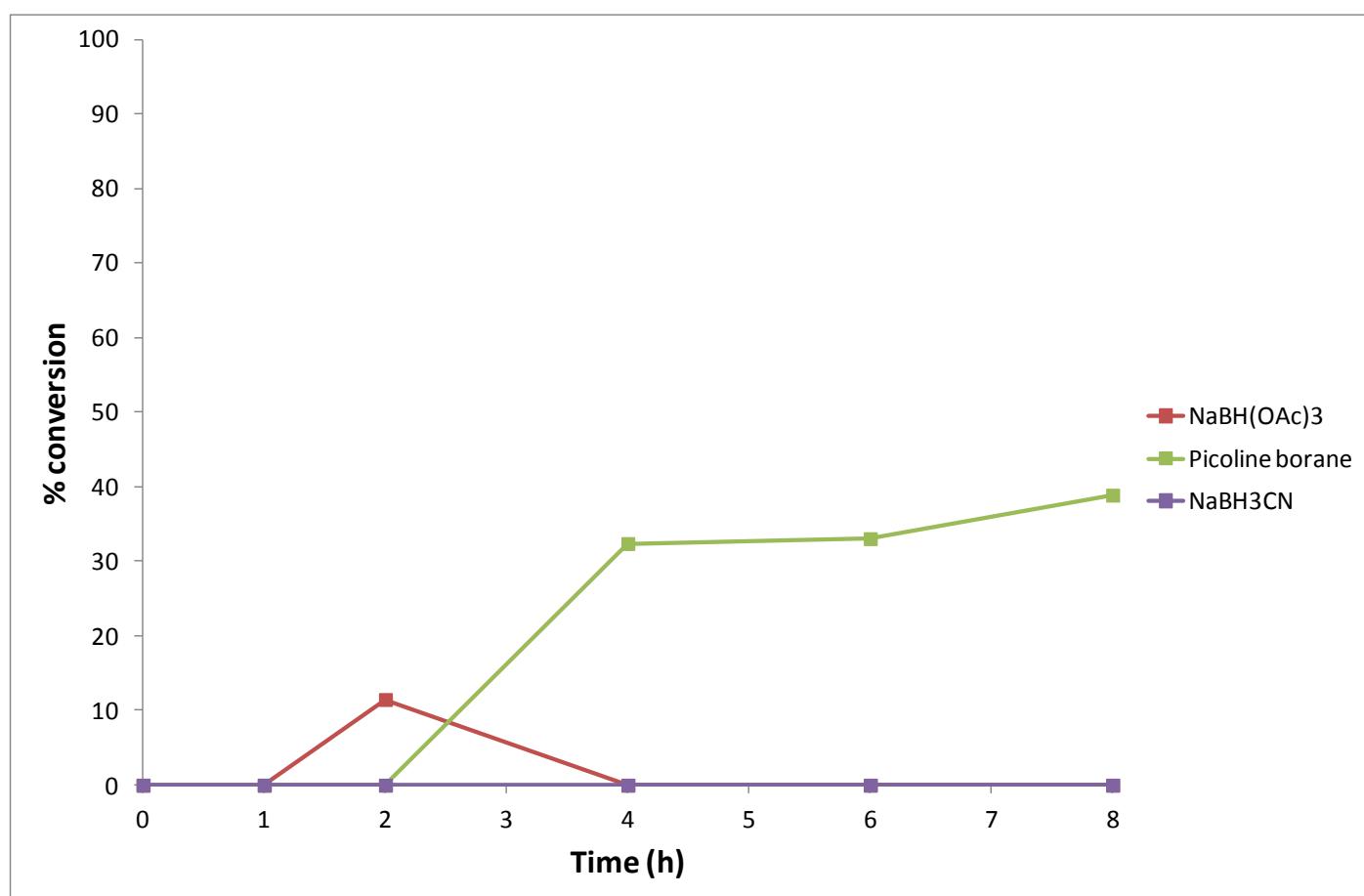
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	48.1	12.2	44.1
1	61.7	24.3	69.2
2	61.7	31.0	71.6
4	61.7	37.2	71.6
6	61.7	43.7	71.6
8	61.7	78.6	71.6
24	61.7	78.6	71.6

Reaction 4: TBME



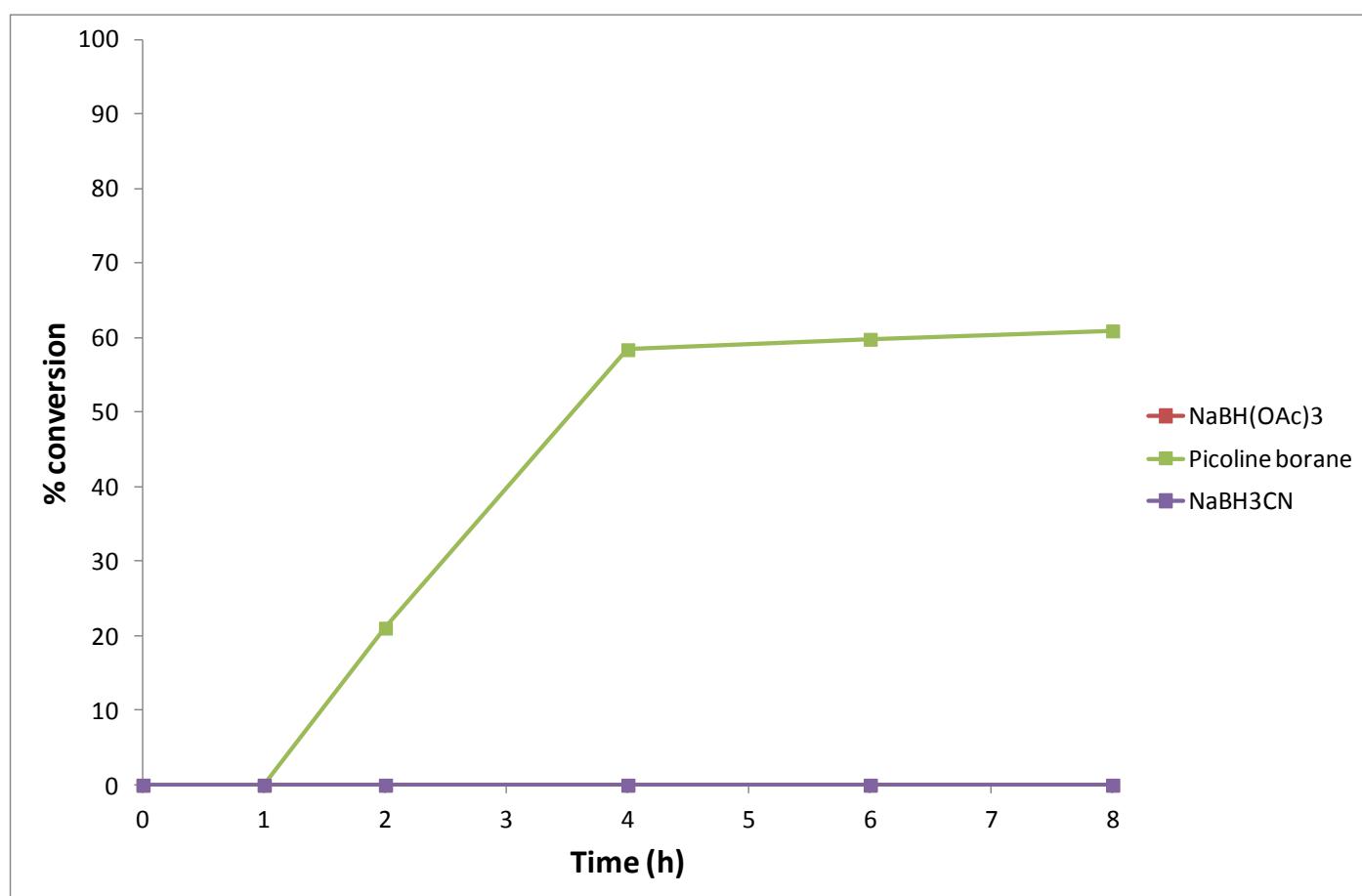
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	0.0	0.0	0.0
1	0.0	0.0	0.0
2	11.7	0.0	0.0
4	0.0	20.9	0.0
6	0.0	19.4	0.0
8	0.0	19.5	0.0
24	0.0	19.5	0.0

Reaction 4: CPME



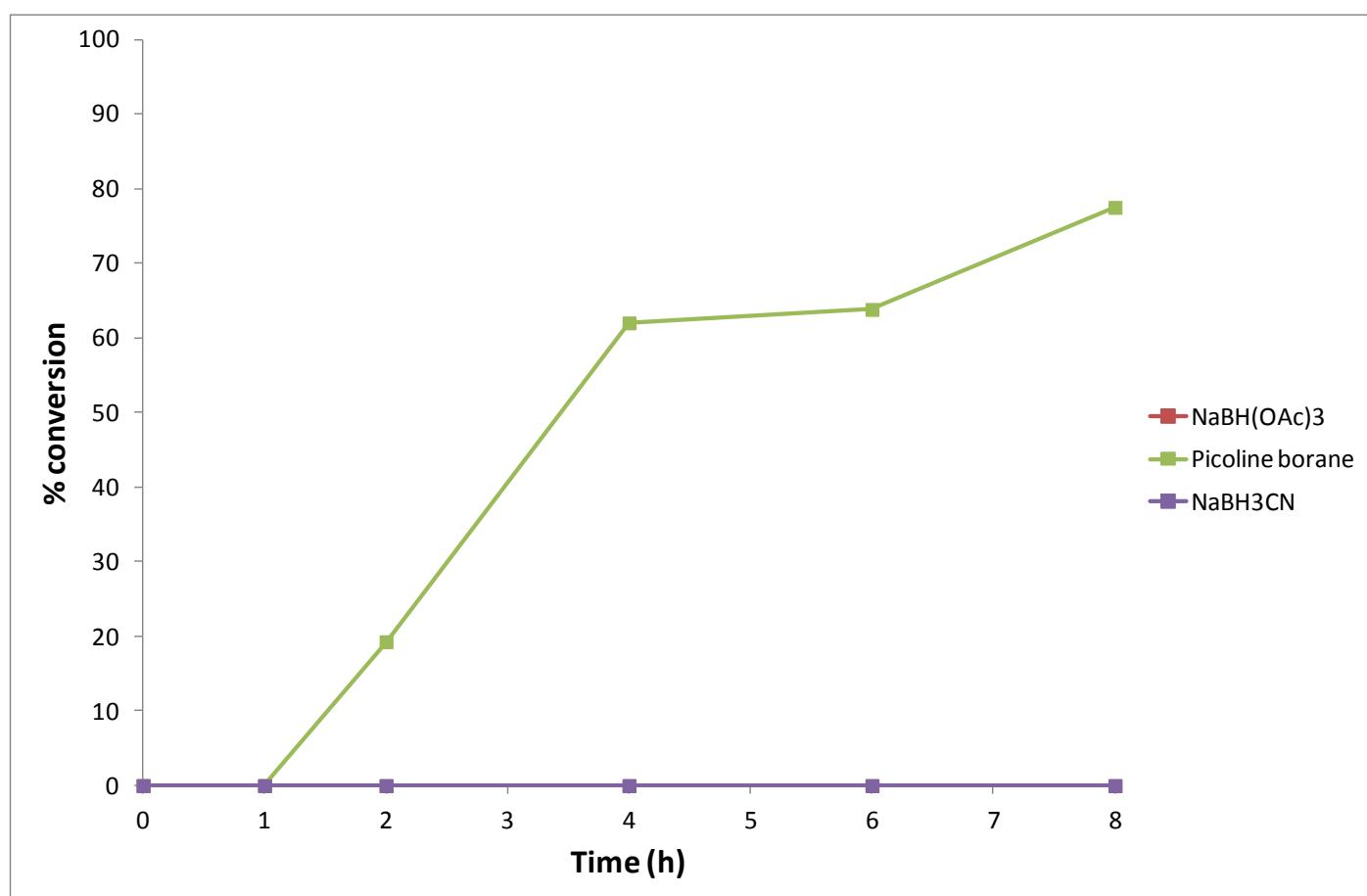
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	0.0	0.0	0.0
1	0.0	0.0	0.0
2	11.5	0.0	0.0
4	0.0	32.4	0.0
6	0.0	33.1	0.0
8	0.0	38.9	0.0
24	0.0	38.9	0.0

Reaction 4: DCE



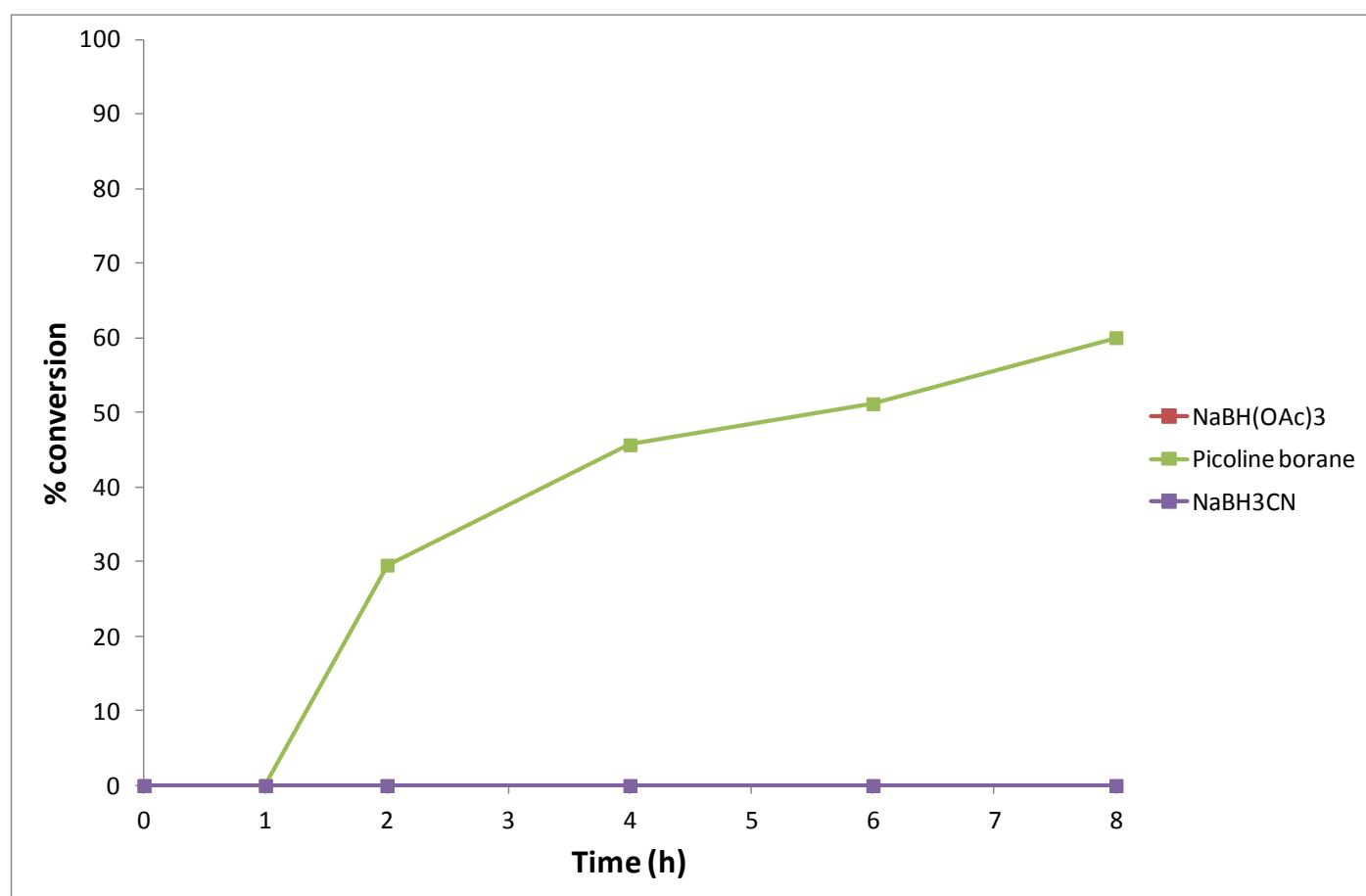
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	0.0	0.0	0.0
1	0.0	0.0	0.0
2	0.0	21.1	0.0
4	0.0	58.4	0.0
6	0.0	59.8	0.0
8	0.0	60.9	0.0
24	0.0	60.9	0.0

Reaction 4: CH₂Cl₂



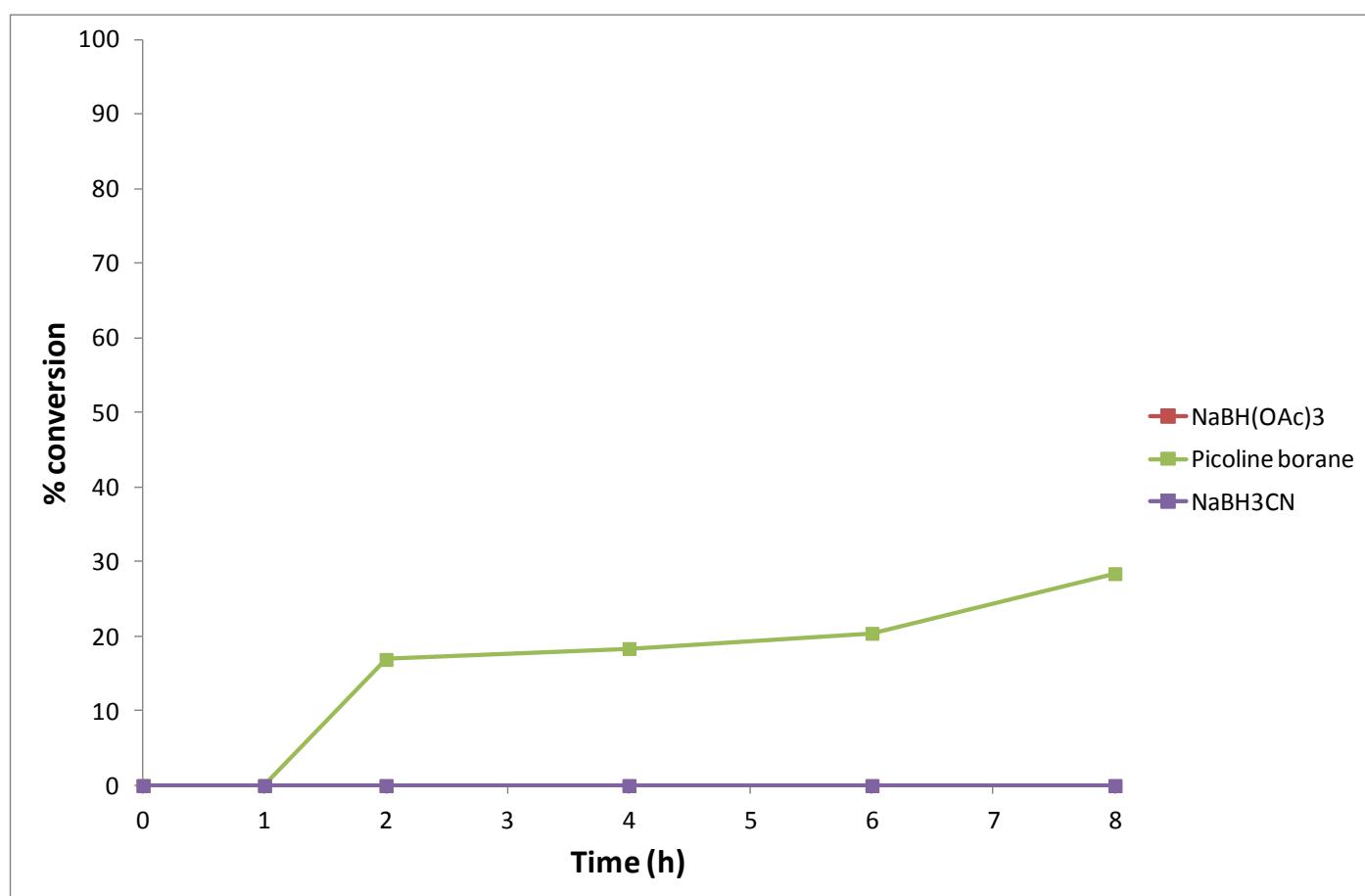
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	0.0	0.0	0.0
1	0.0	0.0	0.0
2	0.0	19.3	0.0
4	0.0	62.0	0.0
6	0.0	63.8	0.0
8	0.0	77.5	0.0
24	0.0	77.5	0.0

Reaction 4: DMC



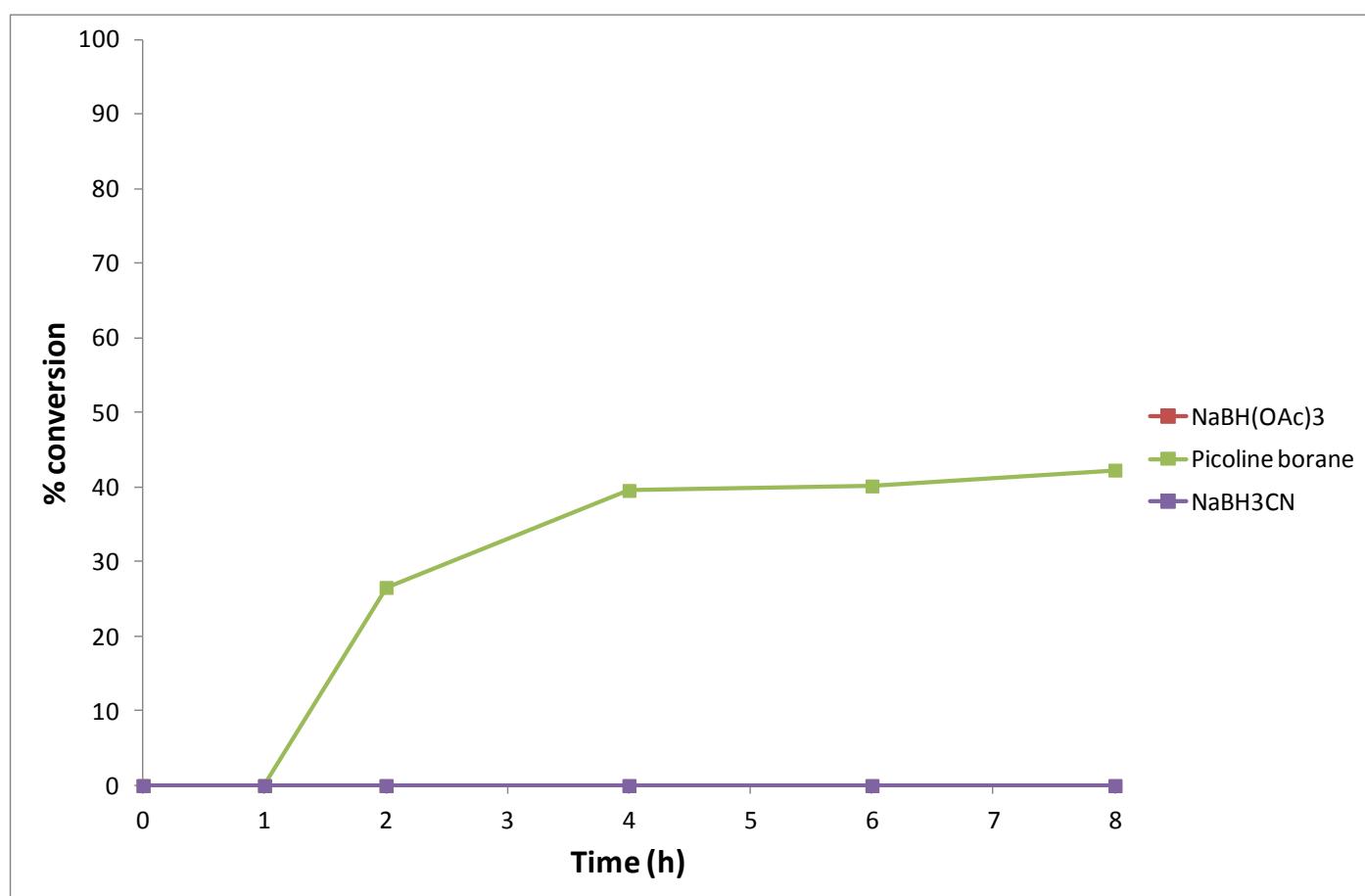
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	0.0	0.0	0.0
1	0.0	0.0	0.0
2	0.0	29.6	0.0
4	0.0	45.7	0.0
6	0.0	51.2	0.0
8	0.0	60.0	0.0
24	0.0	60.0	0.0

Reaction 4: DMF



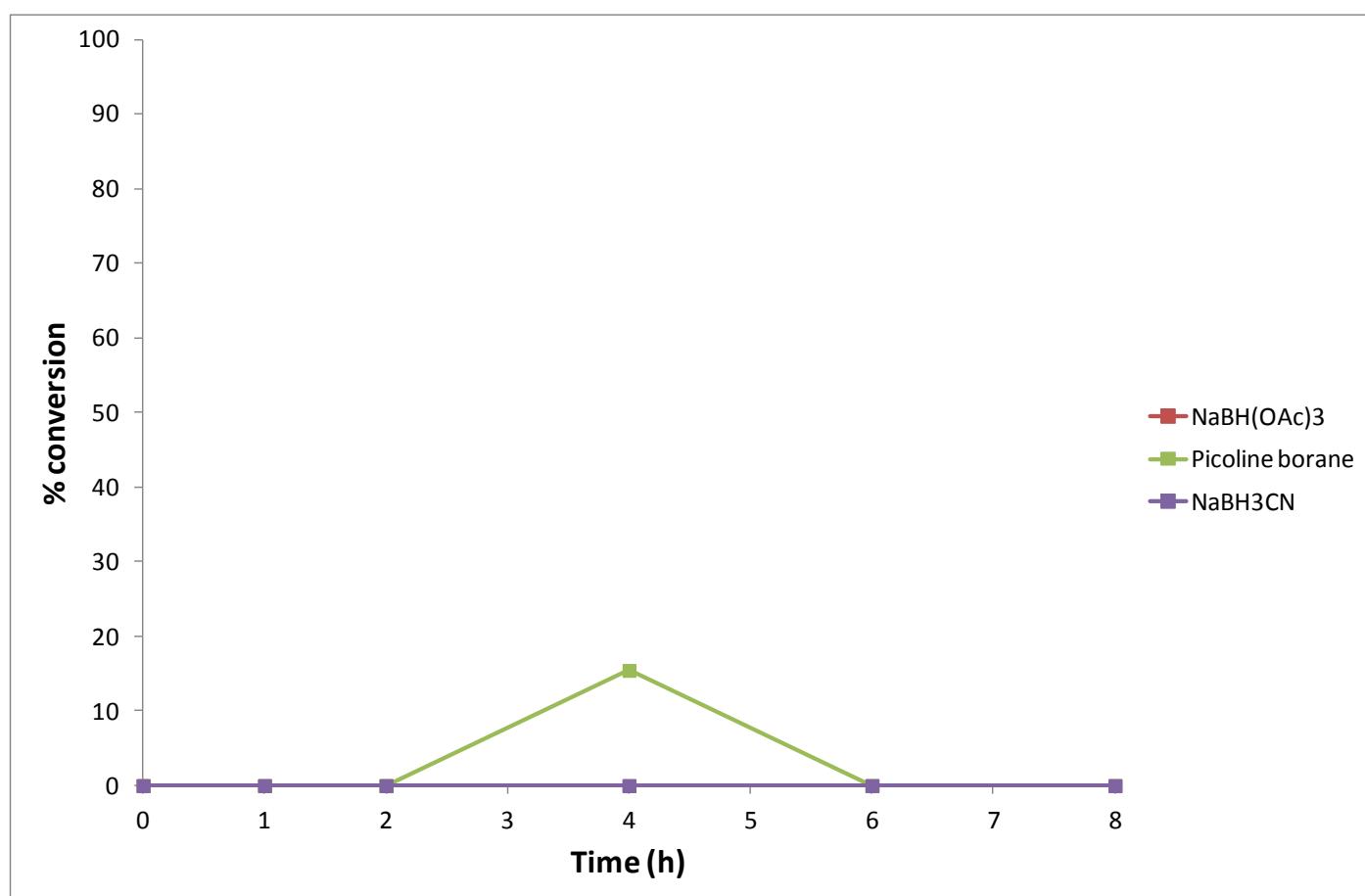
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	0.0	0.0	0.0
1	0.0	0.0	0.0
2	0.0	16.9	0.0
4	0.0	18.4	0.0
6	0.0	20.4	0.0
8	0.0	28.4	0.0
24	0.0	28.4	0.0

Reaction 4: EtOAc



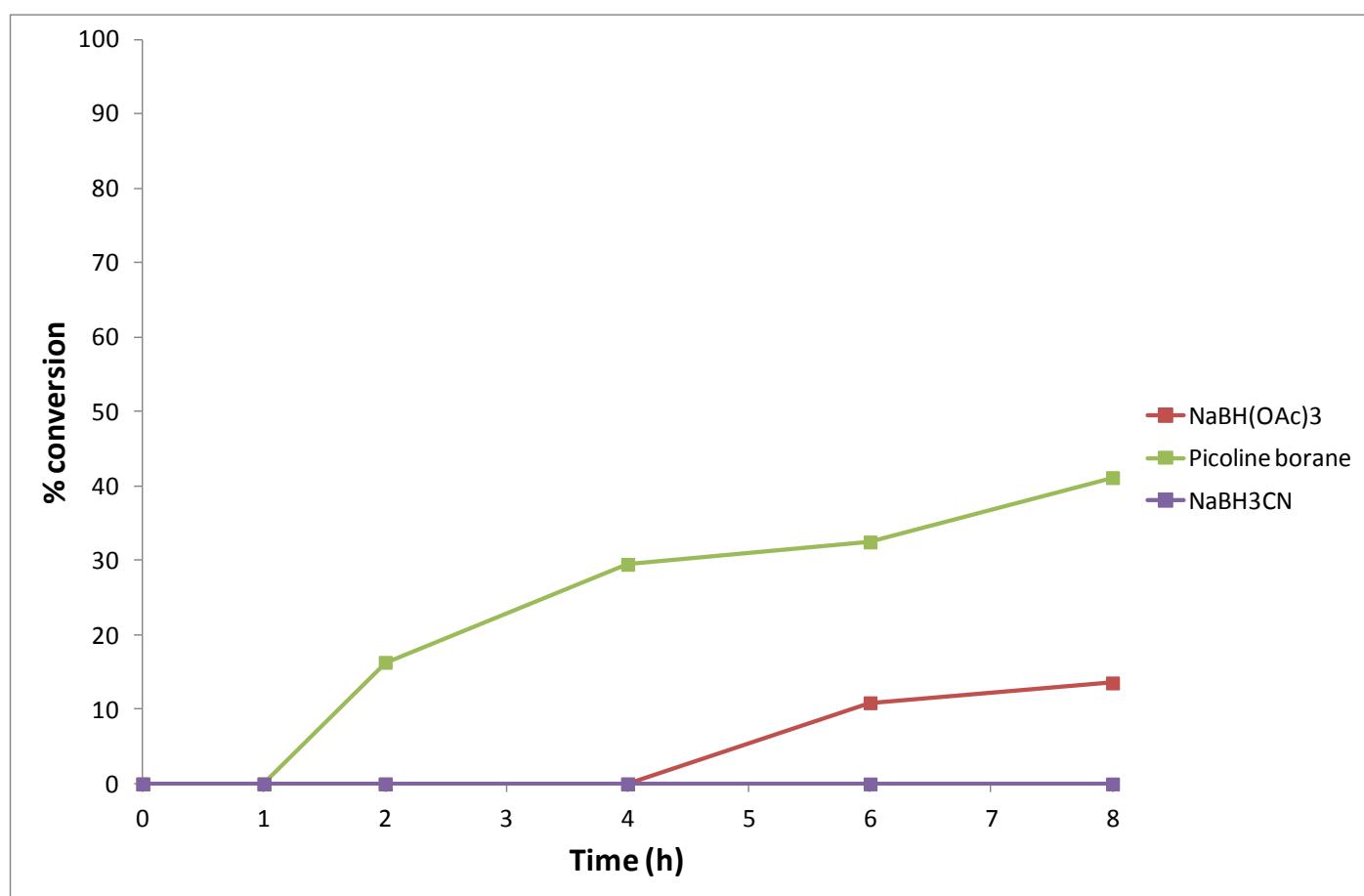
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	0.0	0.0	0.0
1	0.0	0.0	0.0
2	0.0	26.6	0.0
4	0.0	39.6	0.0
6	0.0	40.2	0.0
8	0.0	42.3	0.0
24	0.0	42.3	0.0

Reaction 4: IPA



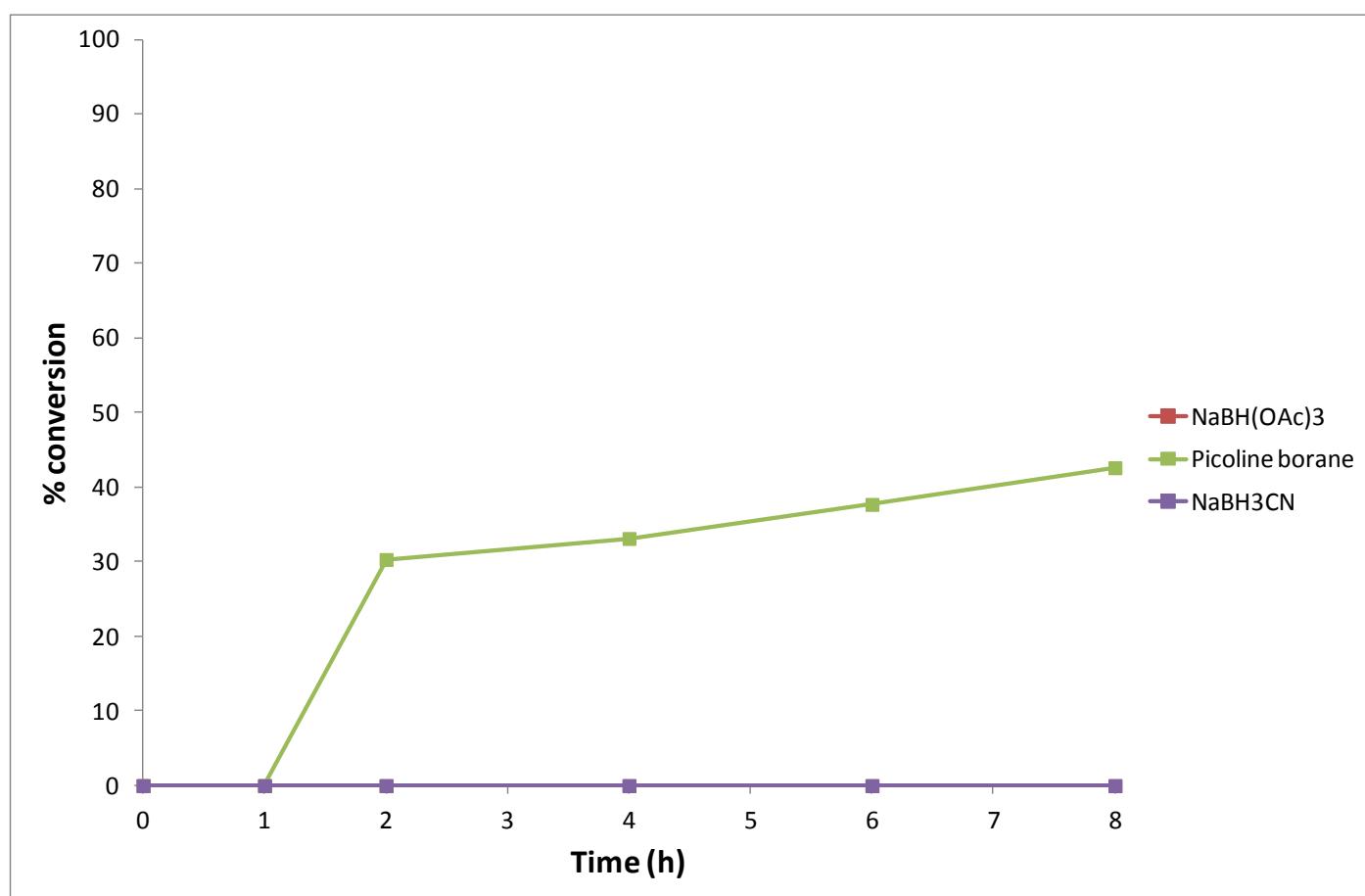
Time (h) / Reducing agent	$\text{NaBH}(\text{OAc})_3$	Picoline borane	NaBH_3CN
0	0.0	0.0	0.0
1	0.0	0.0	0.0
2	0.0	0.0	0.0
4	0.0	15.5	0.0
6	0.0	0.0	0.0
8	0.0	0.0	0.0
24	0.0	0.0	0.0

Reaction 4: 2-MeTHF



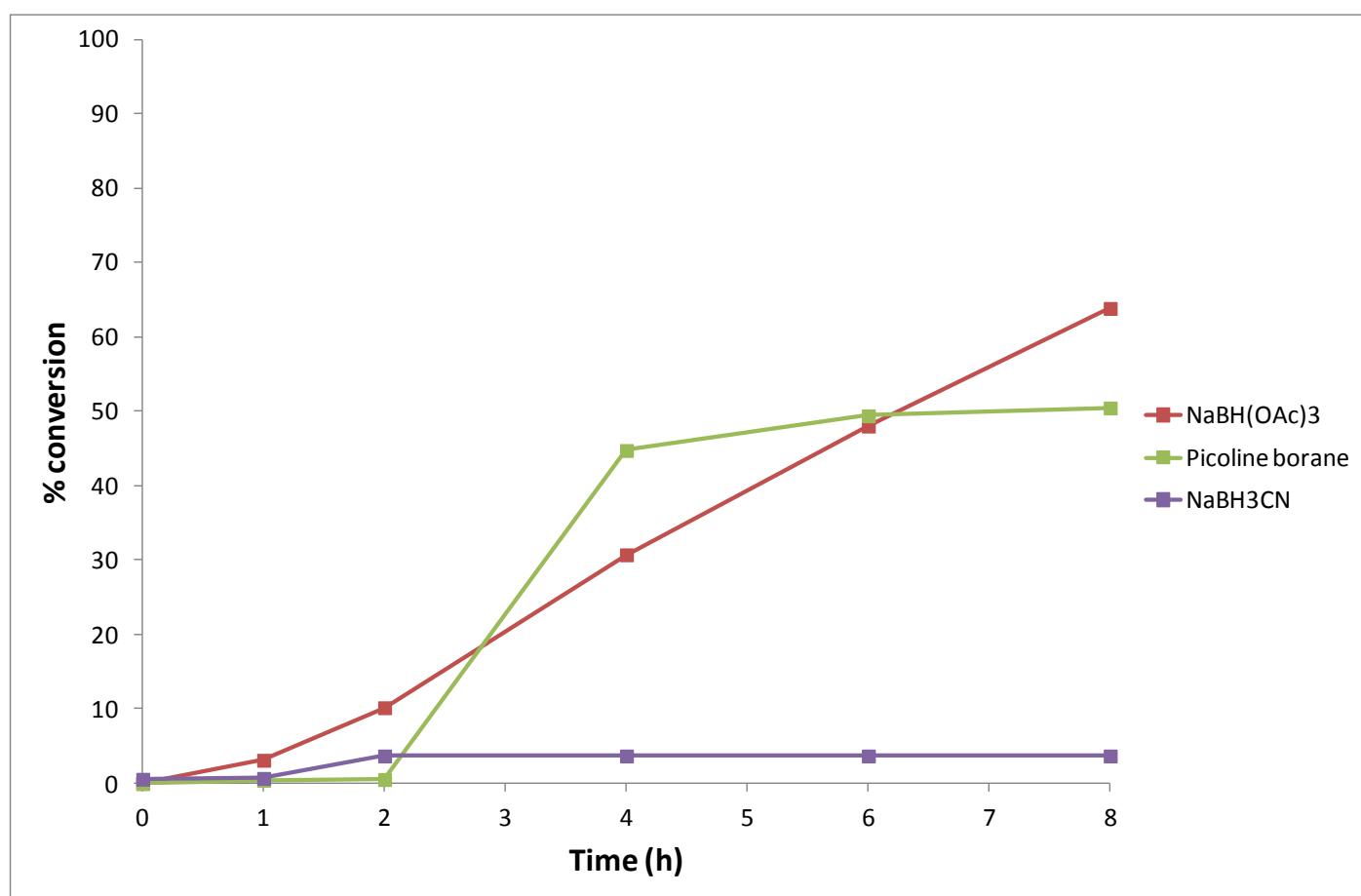
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	0.0	0.0	0.0
1	0.0	0.0	0.0
2	0.0	16.3	0.0
4	0.0	29.5	0.0
6	10.9	32.5	0.0
8	13.6	41.1	0.0
24	12.6	41.1	0.0

Reaction 4: THF



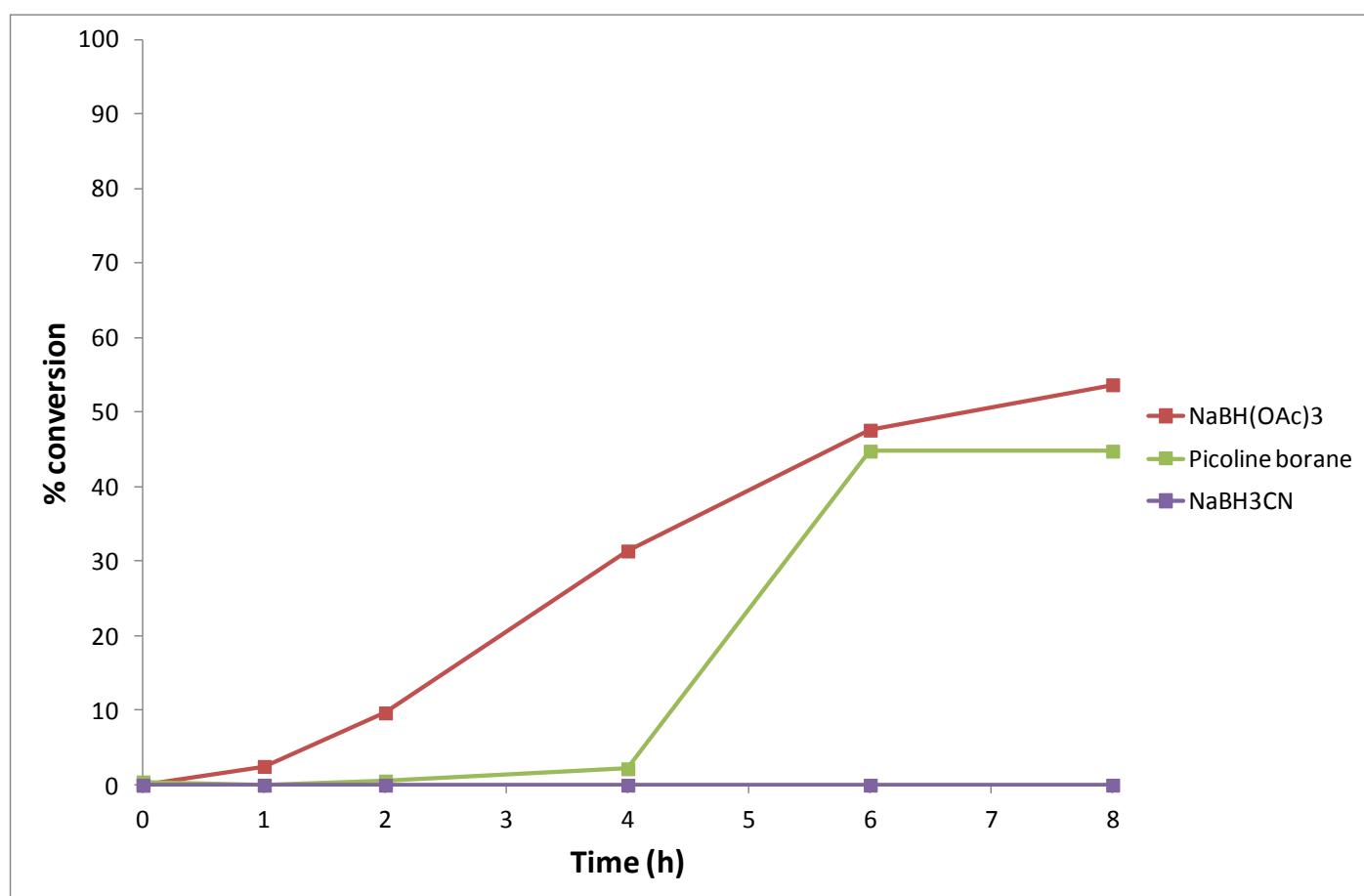
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	0.0	0.0	0.0
1	0.0	0.0	0.0
2	0.0	30.3	0.0
4	0.0	33.1	0.0
6	0.0	37.7	0.0
8	0.0	42.6	0.0
24	0.0	42.6	0.0

Reaction 5: TBME



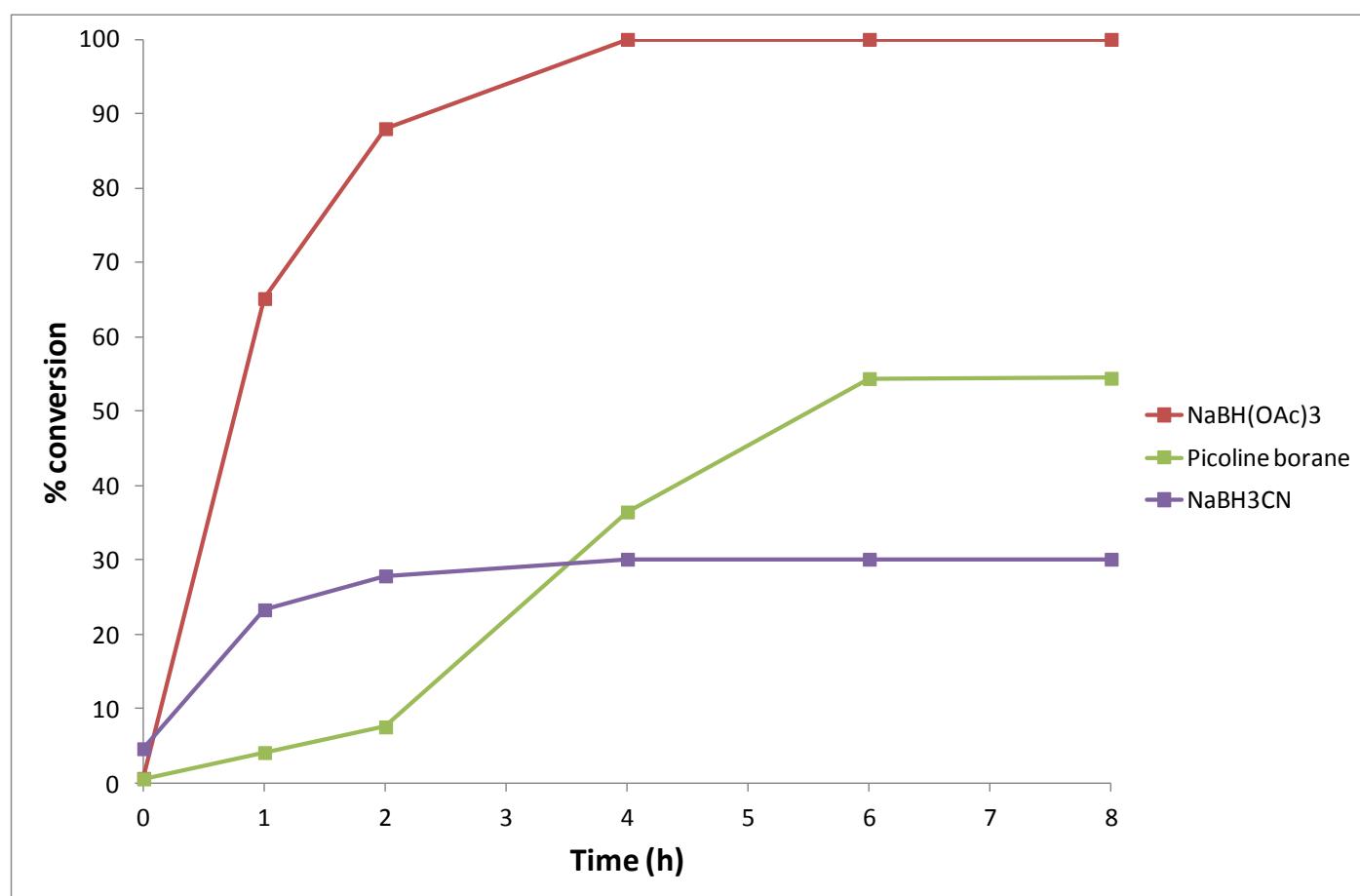
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	0.0	0.0	0.6
1	3.1	0.4	0.7
2	10.2	0.5	3.7
4	30.7	44.7	3.7
6	48.0	49.4	3.7
8	63.8	50.5	3.7
24	89.4	50.5	3.7

Reaction 5: CPME



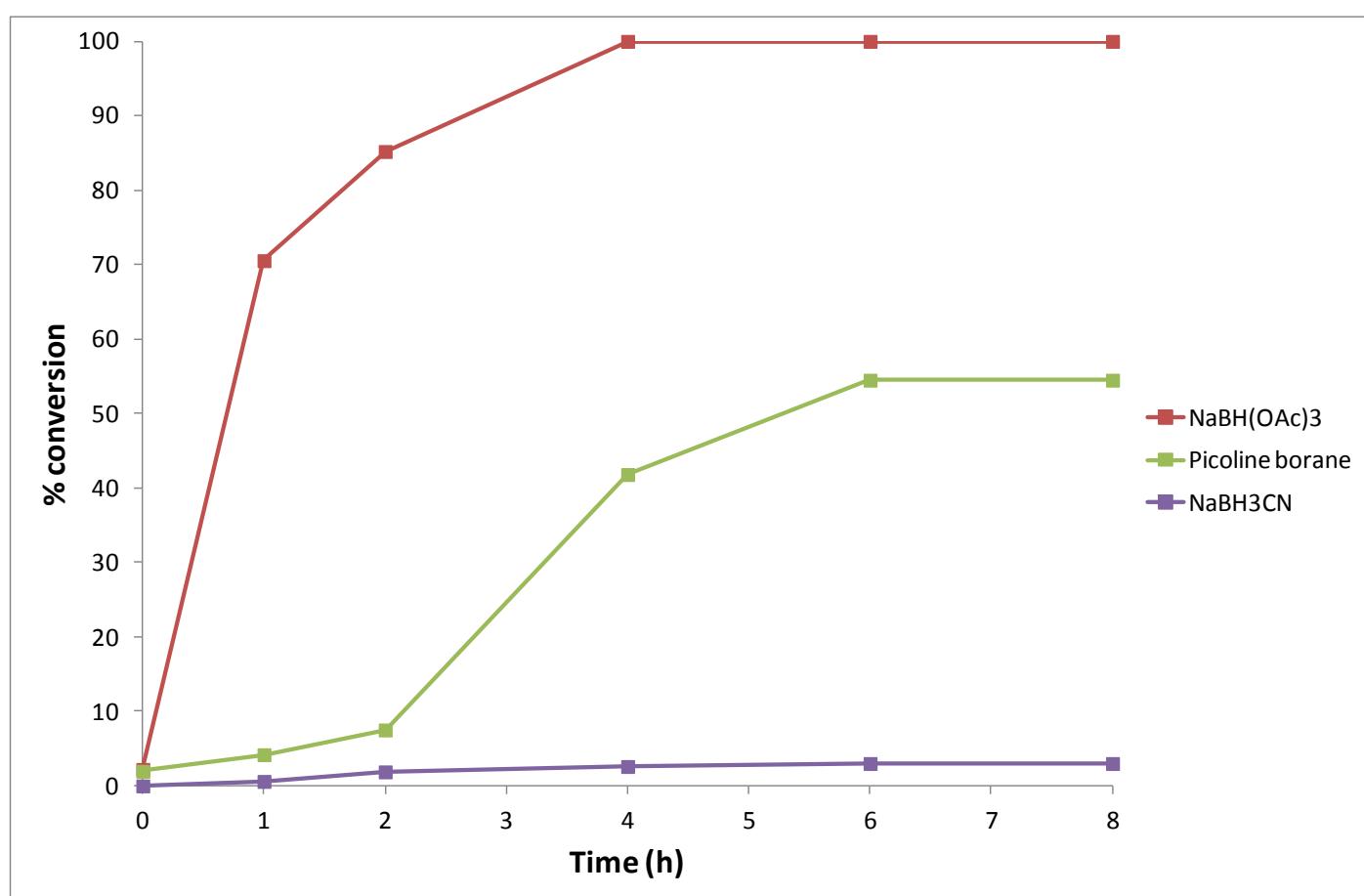
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	0.0	0.4	0.0
1	2.5	0.0	0.0
2	9.7	0.6	0.0
4	31.4	2.2	0.0
6	47.6	44.8	0.0
8	53.7	44.8	0.0
24	87.4	44.8	0.0

Reaction 5: DCE



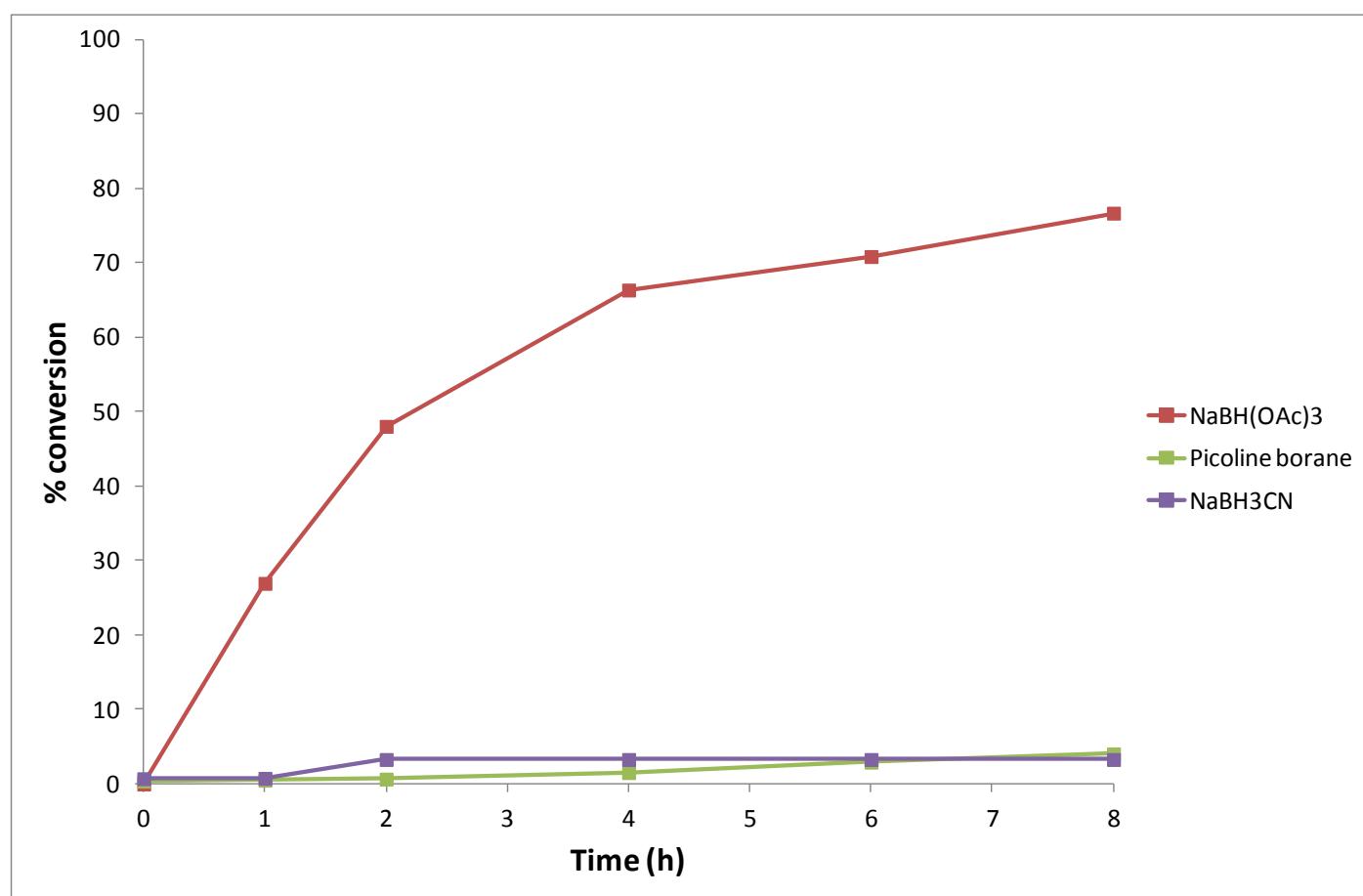
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	0.7	2.0	4.7
1	65.2	4.2	23.3
2	88.0	7.5	27.9
4	100.0	41.8	30.1
6	100.0	54.5	30.1
8	100.0	54.5	30.1
24	100.0	54.5	30.1

Reaction 5: CH₂Cl₂



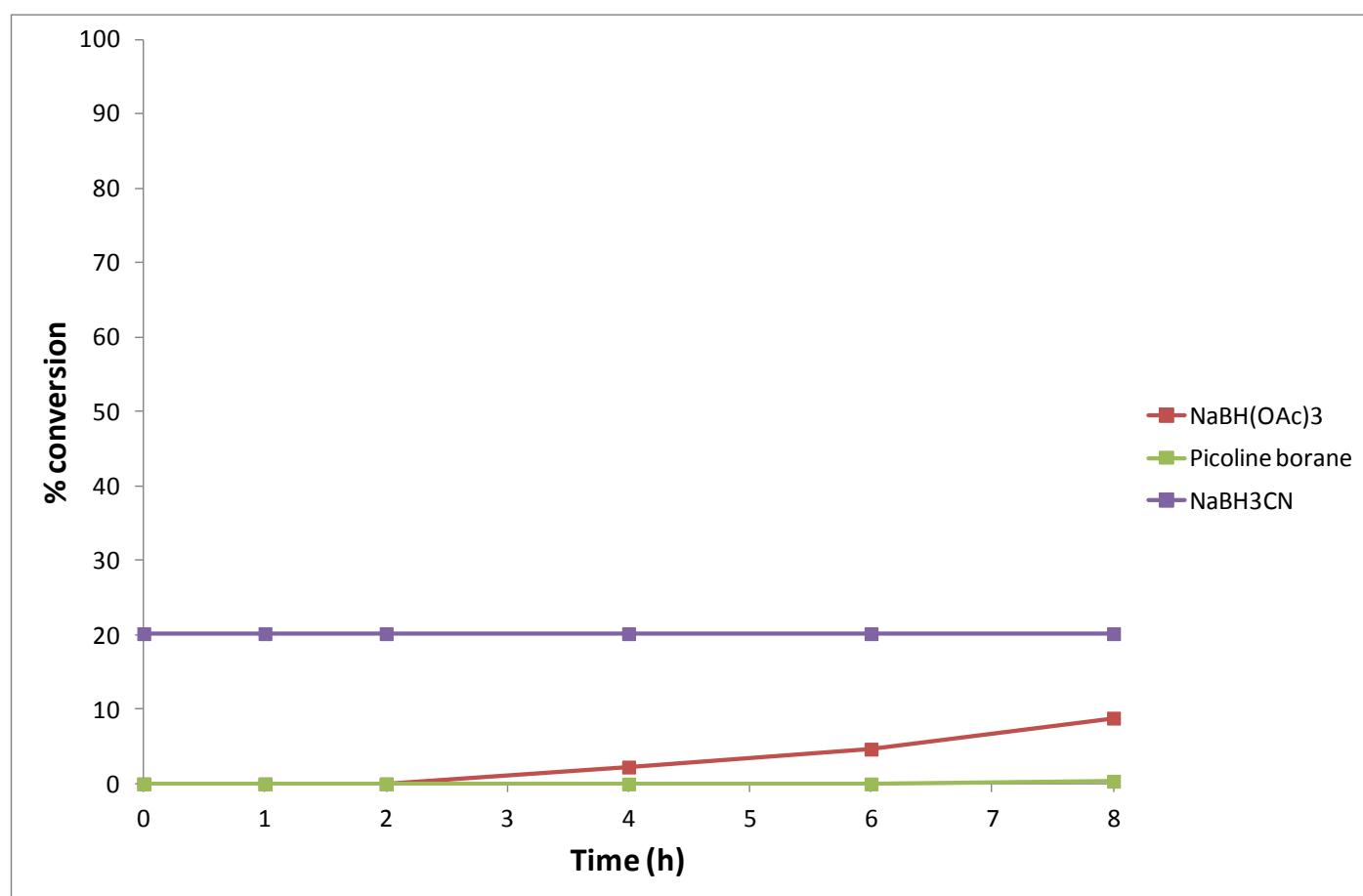
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	2.2	2.0	0.0
1	70.5	4.2	0.6
2	85.2	7.5	1.9
4	100.0	41.8	2.6
6	100.0	54.5	3.0
8	100.0	54.5	3.0
24	100.0	54.5	3.0

Reaction 5: DMC



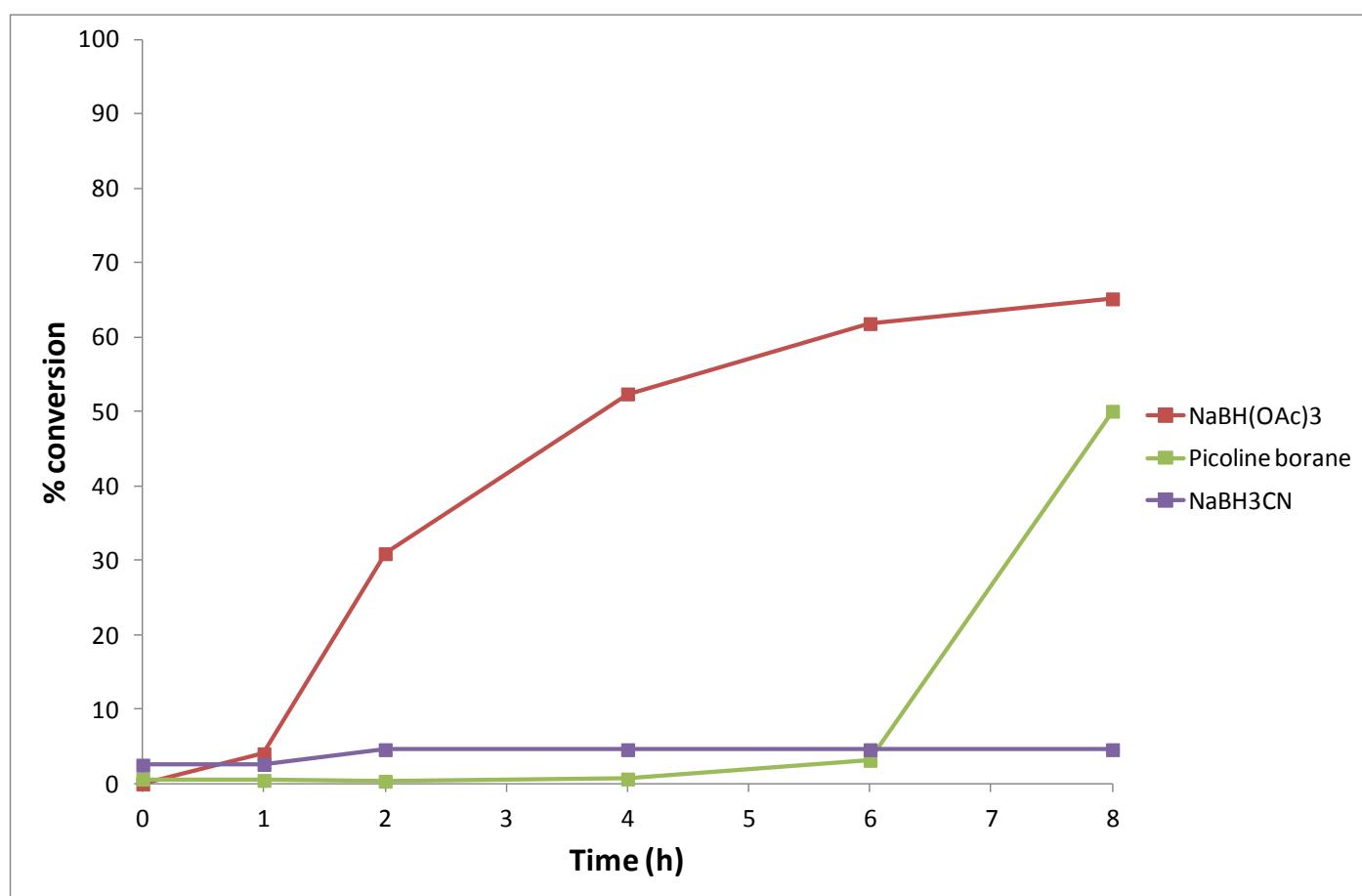
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	0.0	0.4	0.7
1	26.9	0.5	0.8
2	48.0	0.7	3.3
4	66.3	1.5	3.3
6	70.8	2.9	3.3
8	76.7	4.0	3.3
24	87.1	47.8	3.3

Reaction 5: DMF



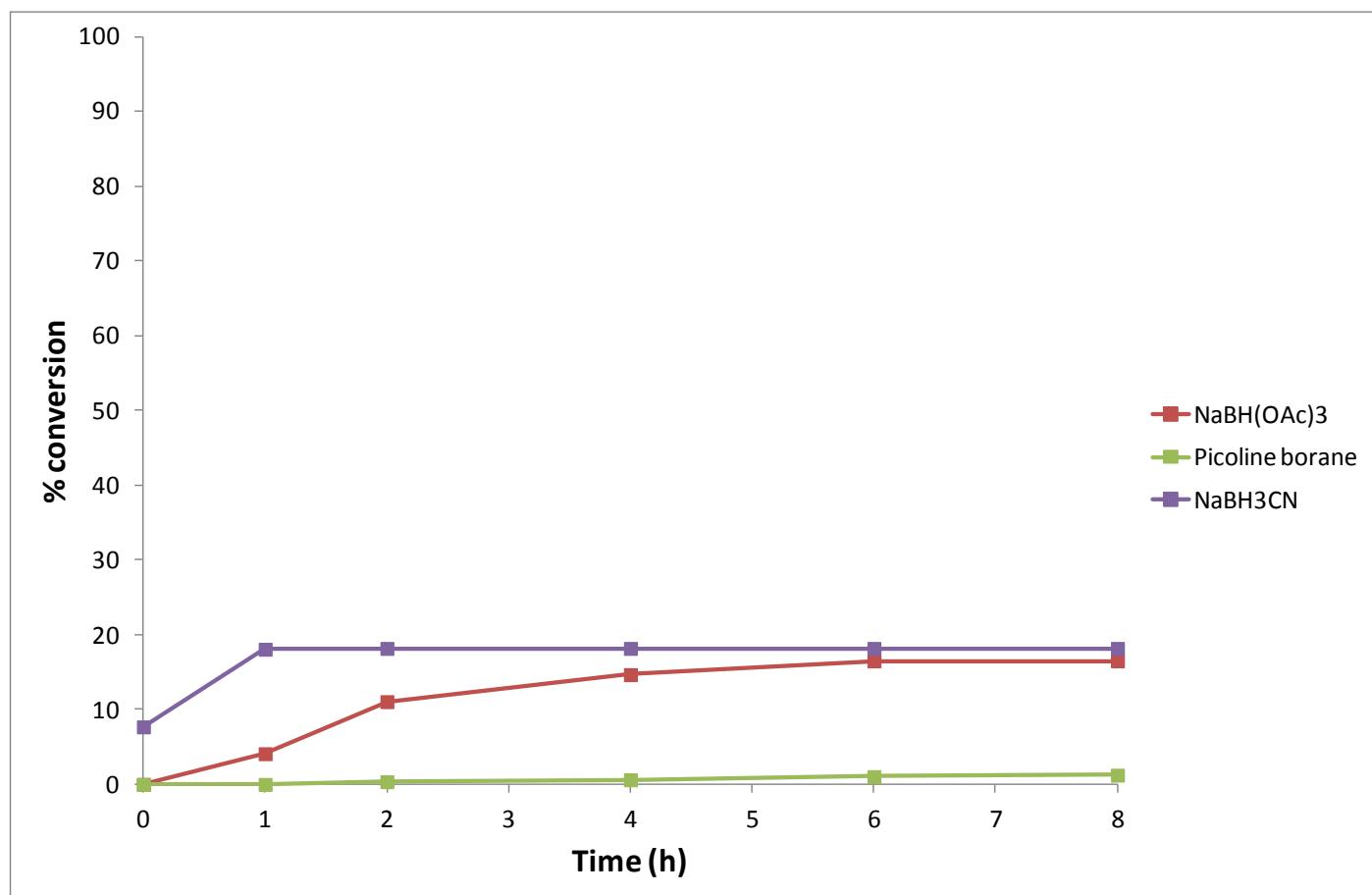
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	0.0	0.0	20.2
1	0.0	0.0	20.2
2	0.0	0.0	20.2
4	2.3	0.0	20.2
6	4.7	0.0	20.2
8	8.8	0.4	20.2
24	25.4	8.6	20.2

Reaction 5: EtOAc



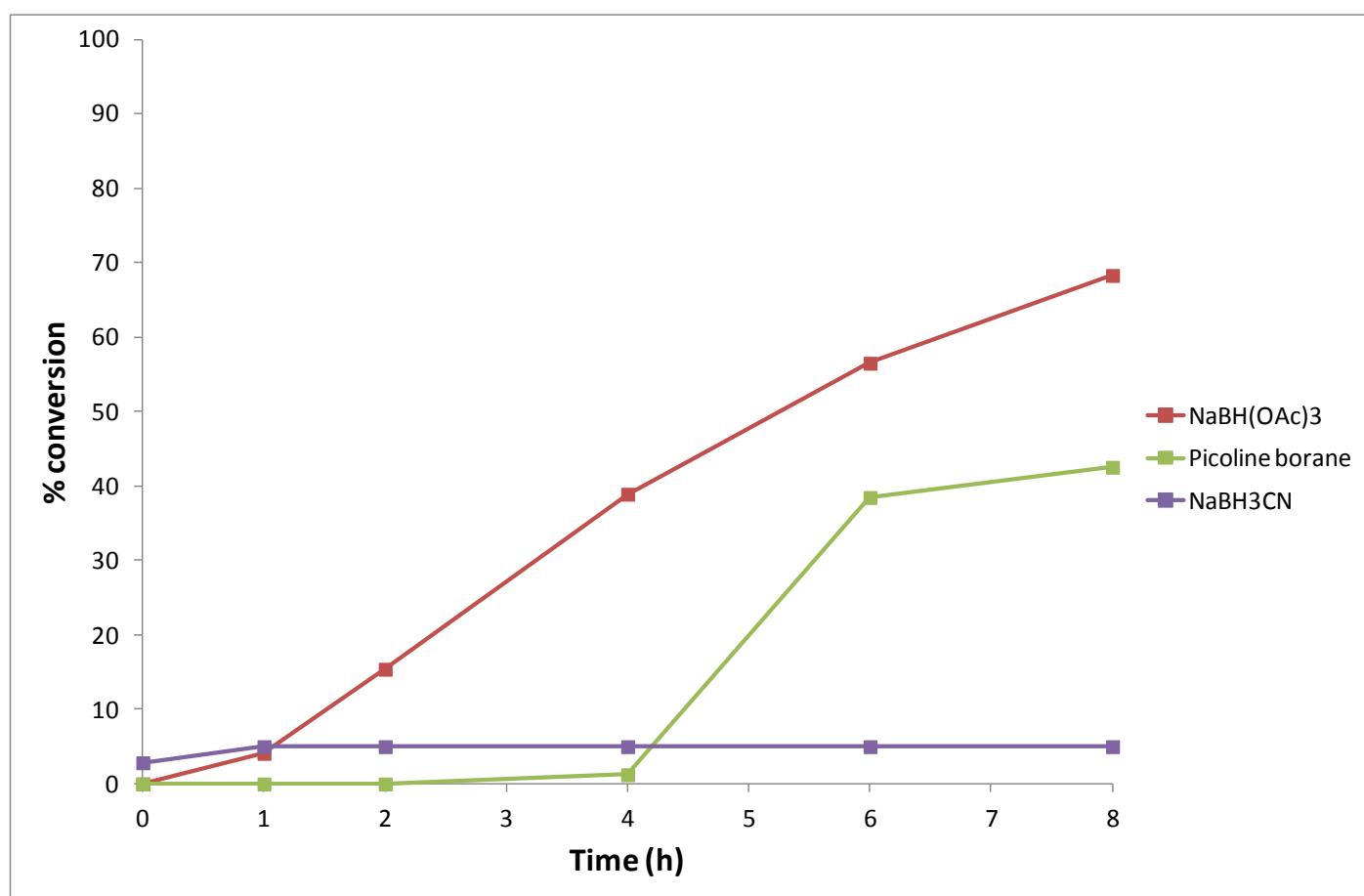
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	0.0	0.6	2.5
1	4.0	0.5	2.6
2	30.9	0.4	4.6
4	52.4	0.7	4.6
6	61.8	3.2	4.6
8	65.2	50.1	4.6
24	72.0	52.3	4.6

Reaction 5: IPA



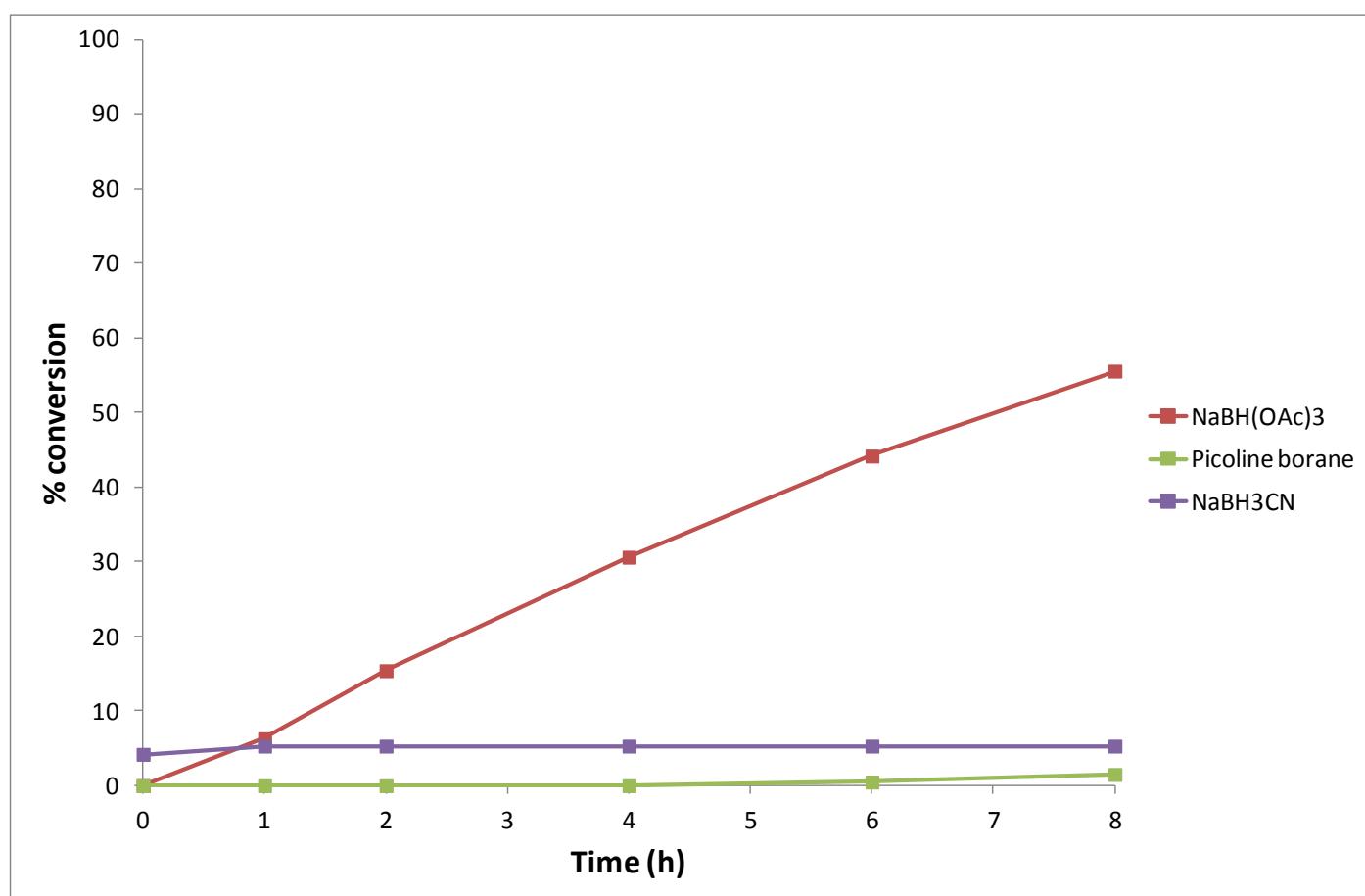
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	0.0	0.0	7.7
1	4.1	0.0	18.1
2	11.1	0.4	18.2
4	14.7	0.6	18.2
6	16.5	1.0	18.2
8	16.5	1.2	18.2
24	17.0	6.1	18.2

Reaction 5: 2-MeTHF



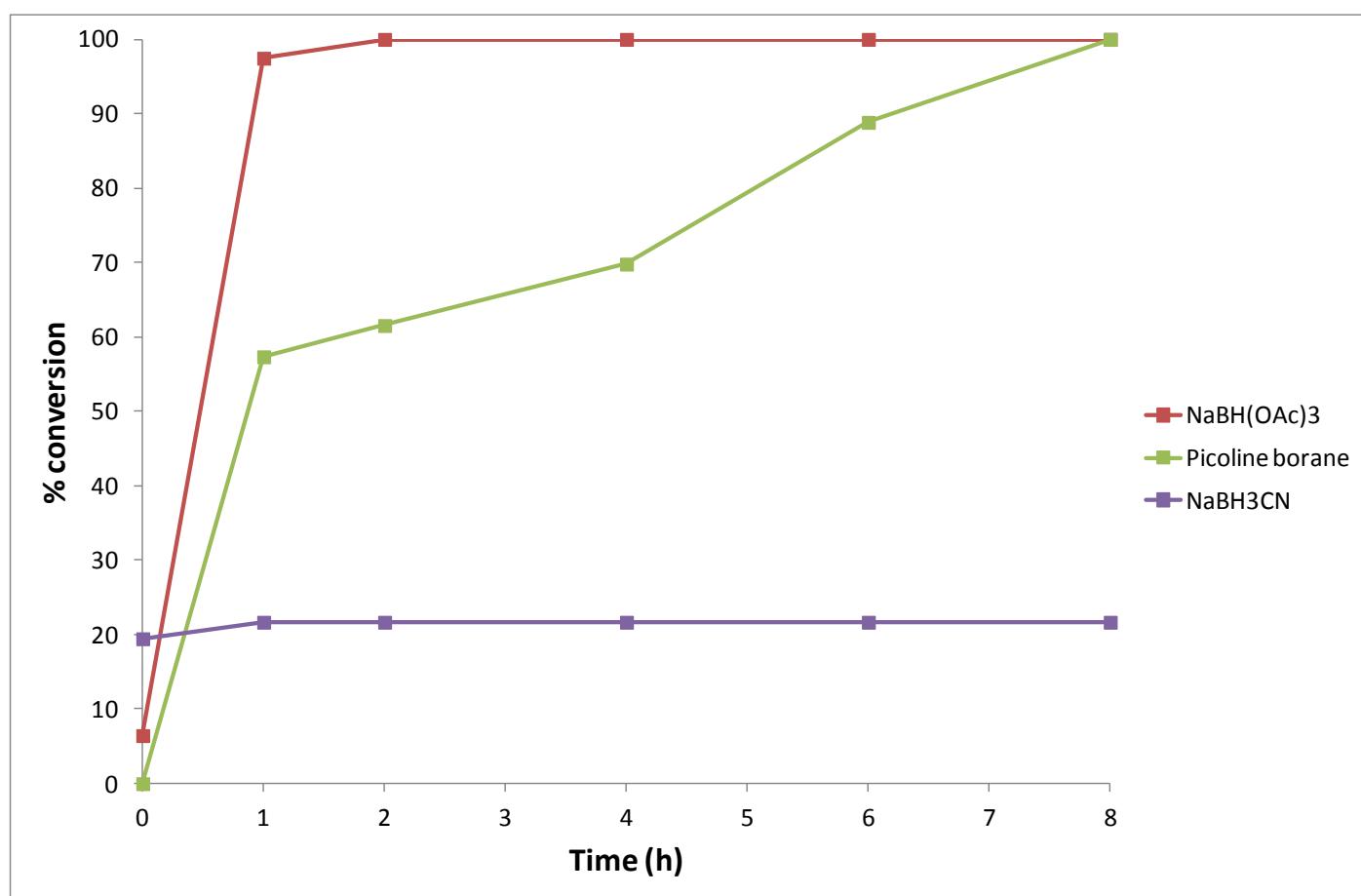
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	0.0	0.0	2.8
1	4.1	0.0	5.0
2	15.4	0.0	5.0
4	38.9	1.2	5.0
6	56.5	38.5	5.0
8	68.3	42.5	5.0
24	73.1	42.5	5.0

Reaction 5: THF



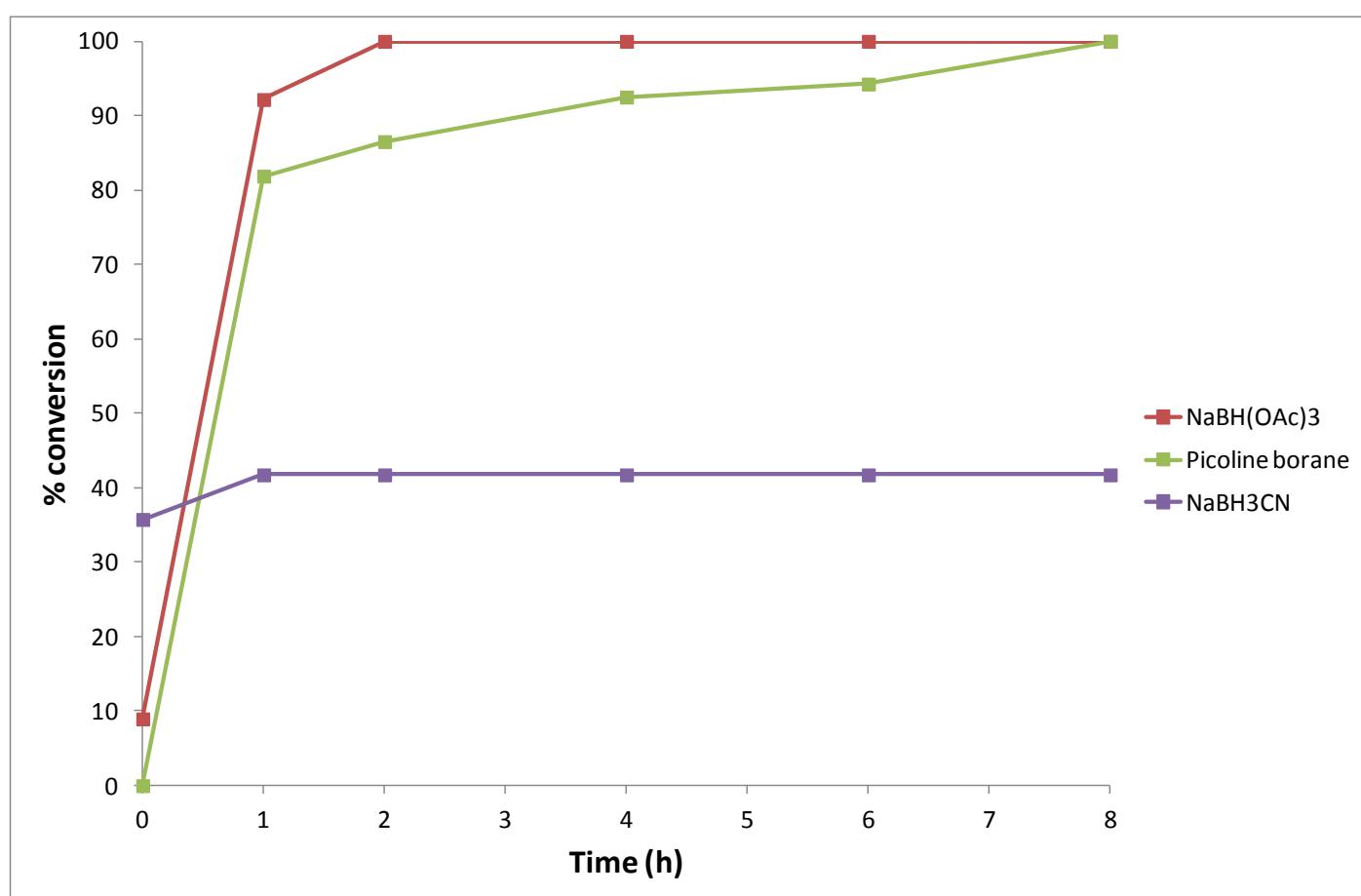
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	0.0	0.0	4.2
1	6.3	0.0	5.3
2	15.4	0.0	5.3
4	30.6	0.0	5.3
6	44.2	0.5	5.3
8	55.6	1.5	5.3
24	57.7	48.9	5.3

Reaction 6: TBME



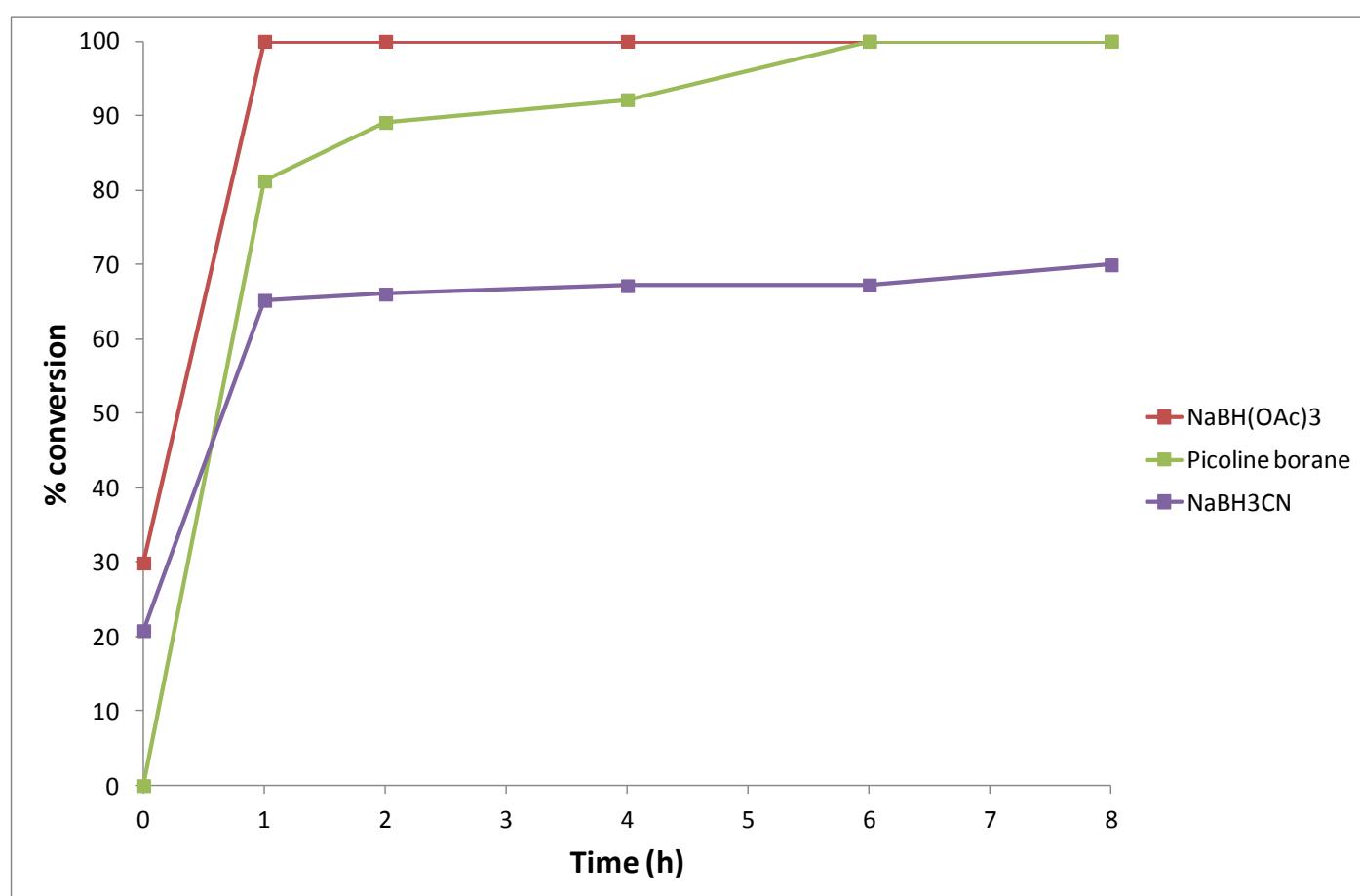
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	6.5	0.0	19.5
1	97.5	57.3	21.7
2	100.0	61.6	21.7
4	100.0	69.8	21.7
6	100.0	88.9	21.7
8	100.0	100.0	21.7
24	100.0	100.0	21.7

Reaction 6: CPME



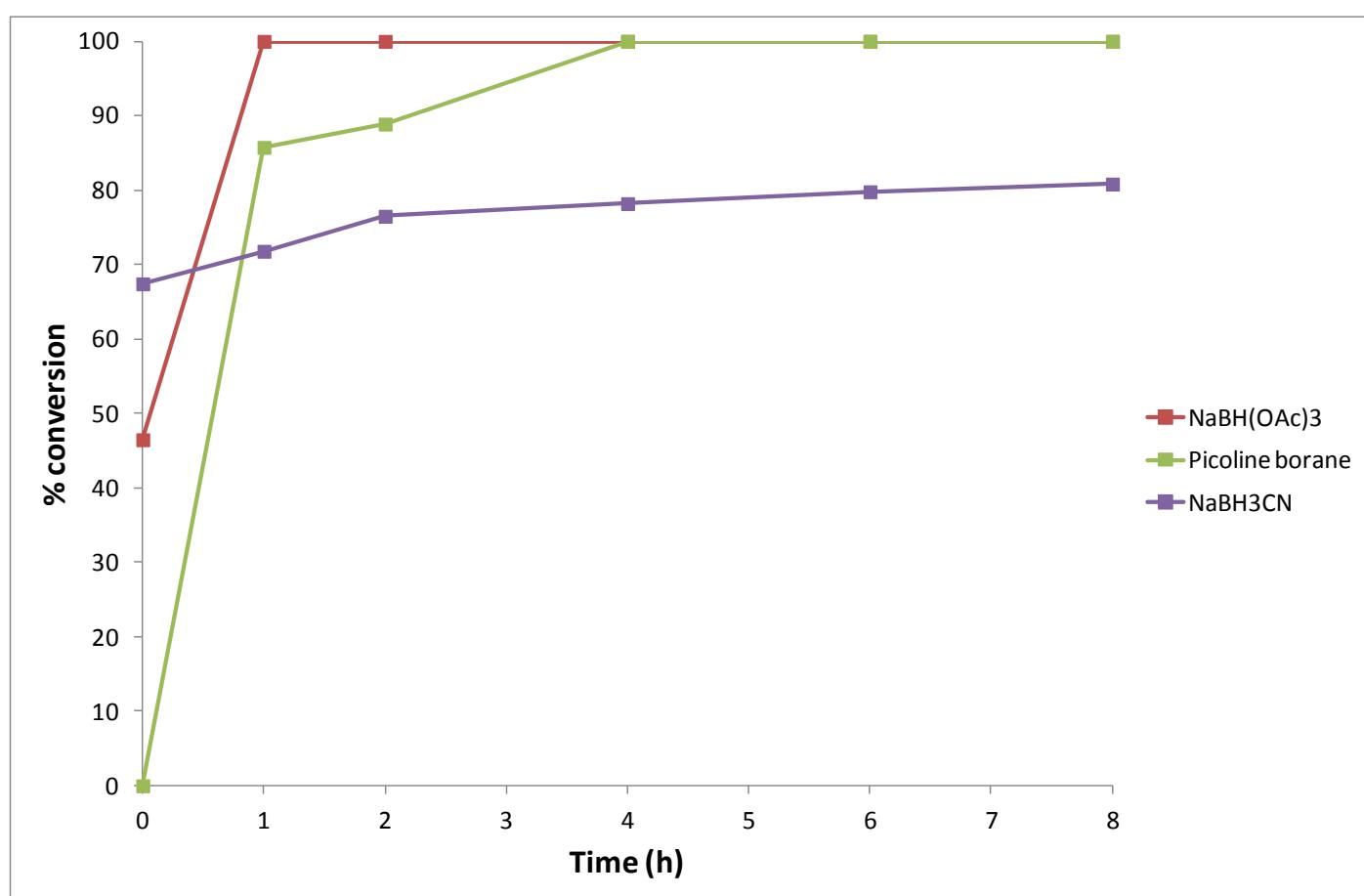
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	9.0	0.0	35.8
1	92.2	81.9	41.7
2	100.0	86.5	41.7
4	100.0	92.5	41.7
6	100.0	94.3	41.7
8	100.0	100.0	41.7
24	100.0	100.0	41.7

Reaction 6: DCE



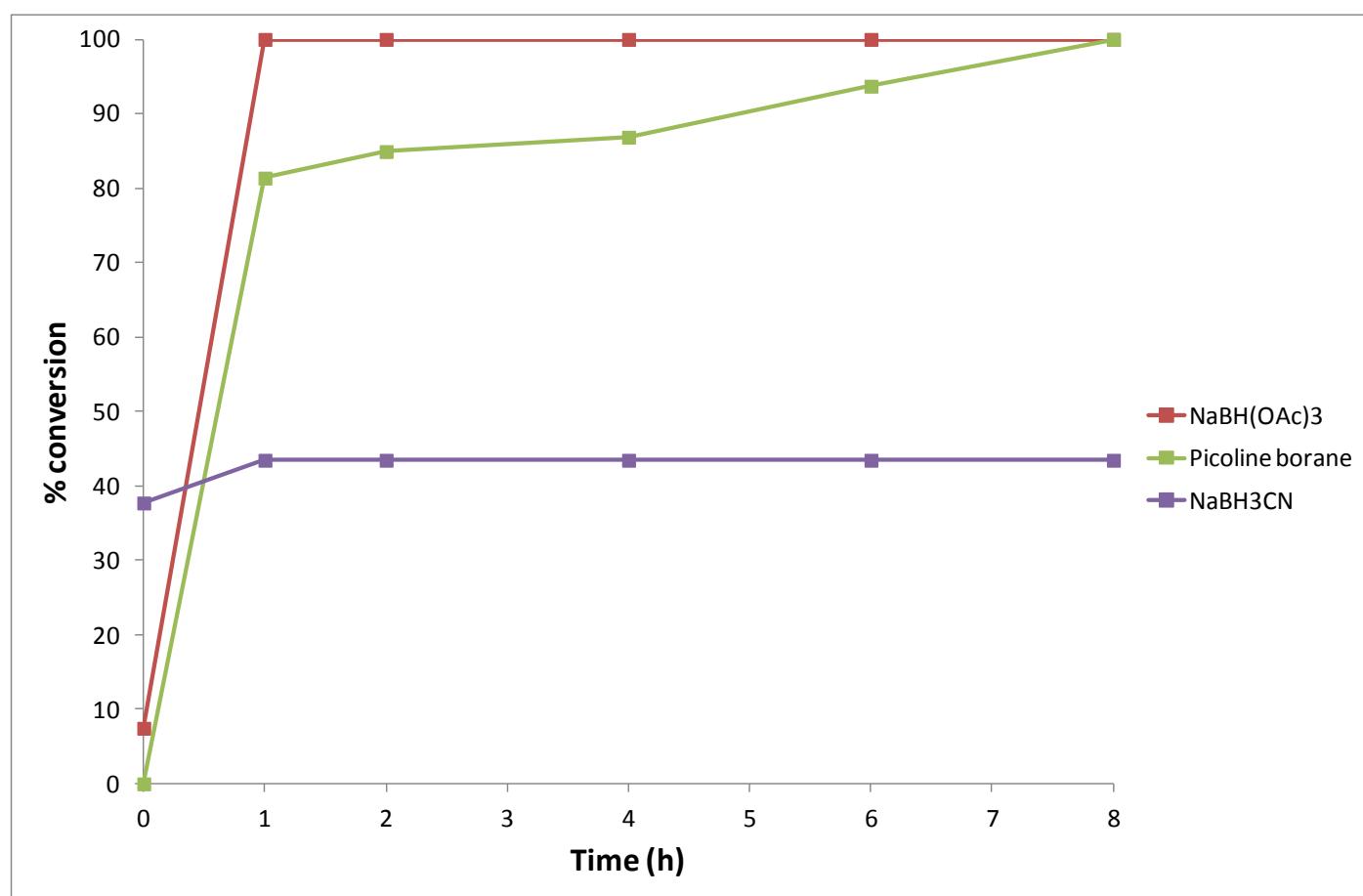
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	29.9	0.0	20.8
1	100.0	81.2	65.2
2	100.0	89.1	66.0
4	100.0	92.2	67.2
6	100.0	100.0	67.3
8	100.0	100.0	70.0
24	100.0	100.0	73.2

Reaction 6: CH₂Cl₂



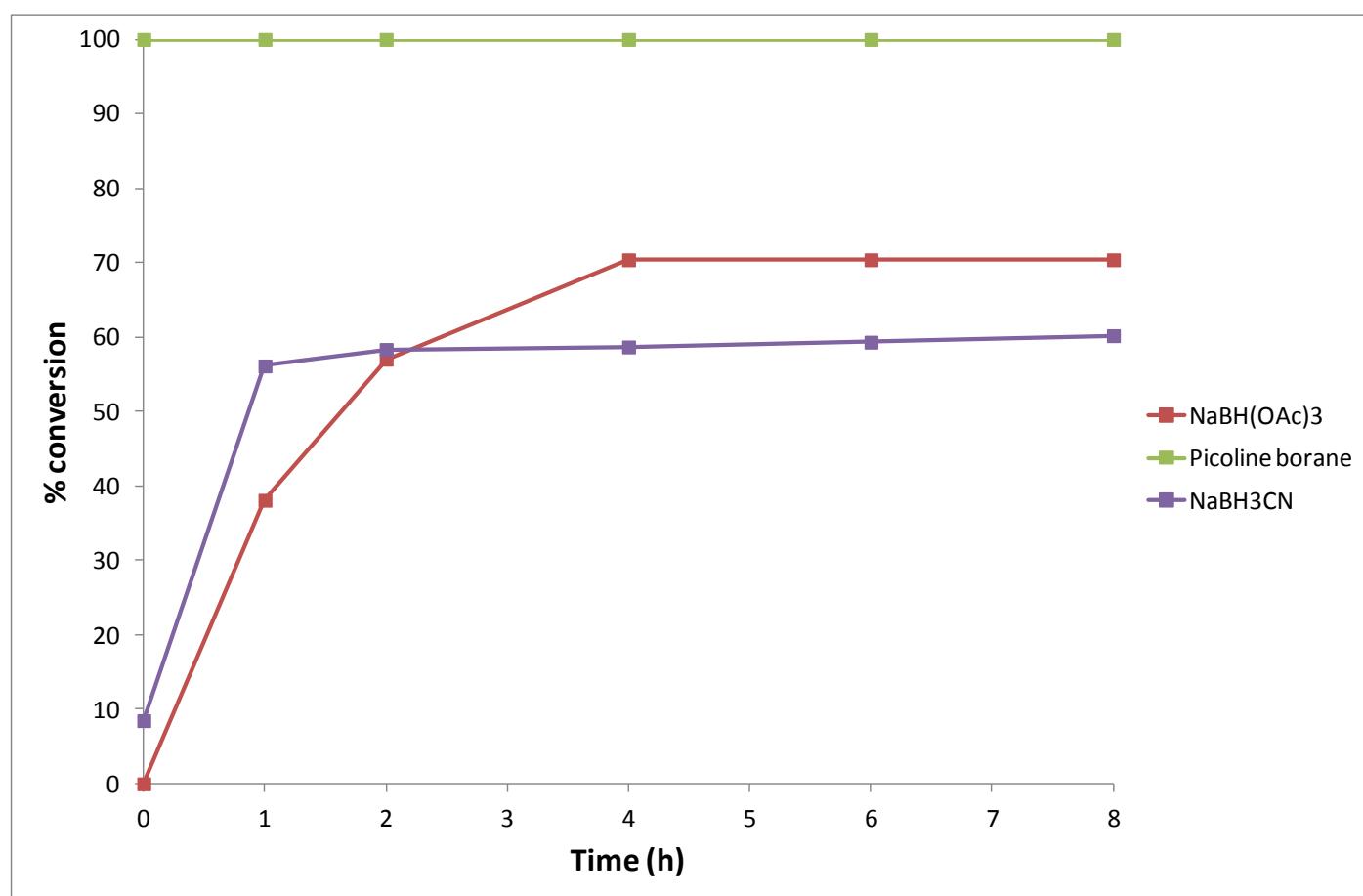
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	46.5	0.0	67.4
1	100.0	85.8	71.8
2	100.0	88.9	76.5
4	100.0	100.0	78.2
6	100.0	100.0	79.8
8	100.0	100.0	80.8
24	100.0	100.0	82.5

Reaction 6: DMC



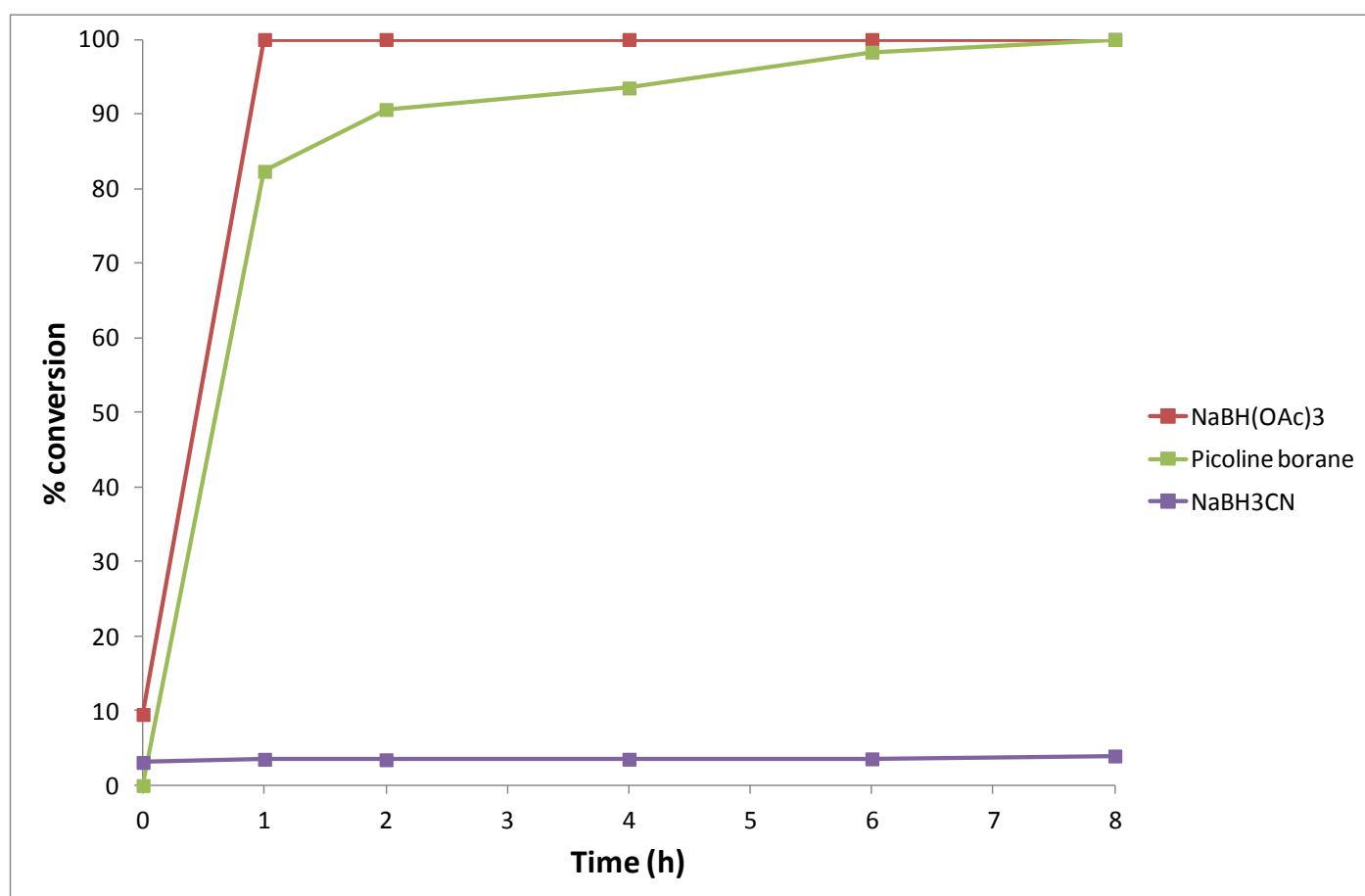
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	7.5	0.0	37.7
1	100.0	81.4	43.5
2	100.0	84.9	43.5
4	100.0	86.9	43.5
6	100.0	93.7	43.5
8	100.0	100.0	43.5
24	100.0	100.0	43.5

Reaction 6: DMF



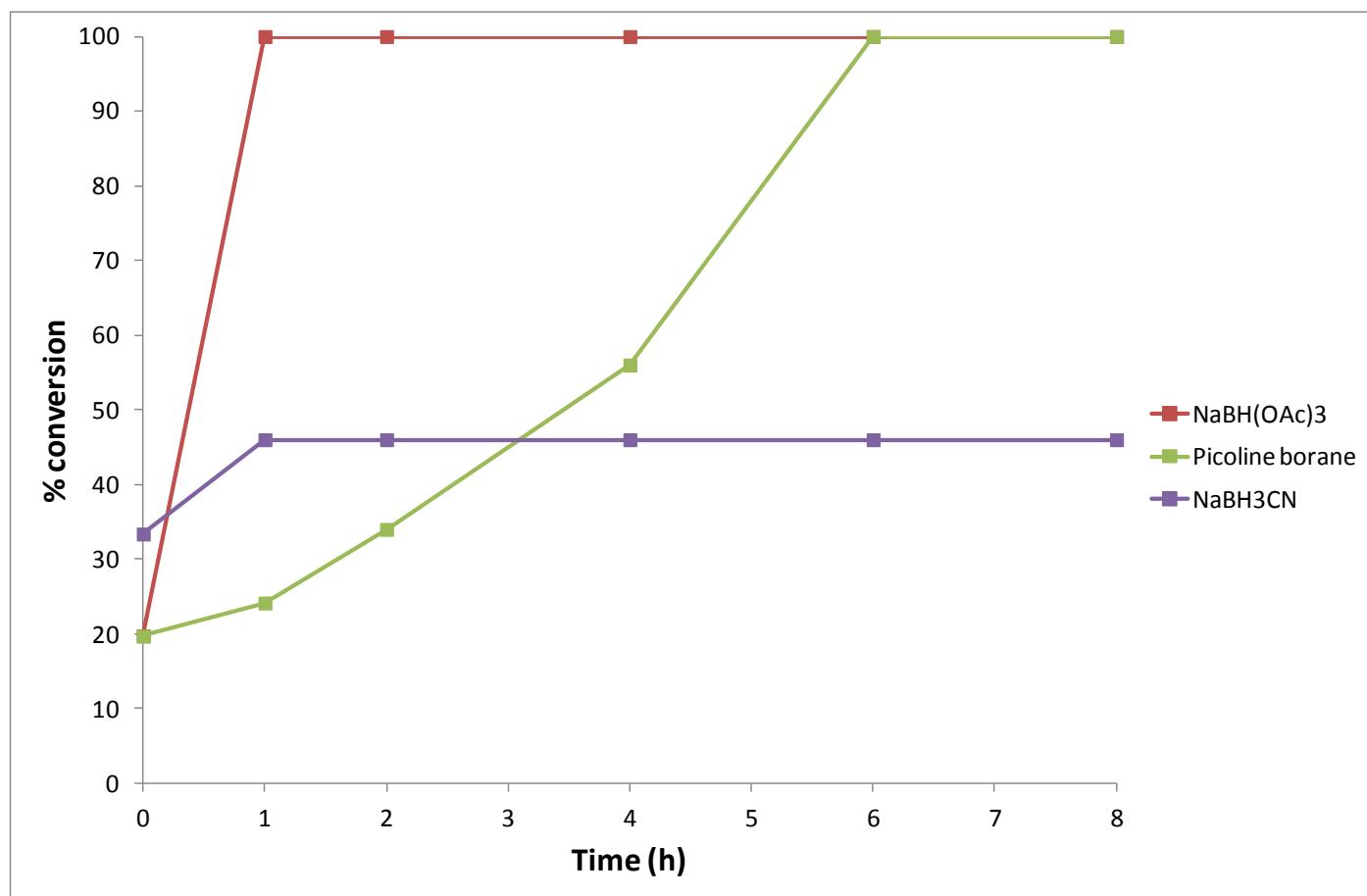
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	0.0	100.0	8.5
1	38.1	100.0	56.1
2	57.0	100.0	58.3
4	70.4	100.0	58.7
6	70.4	100.0	59.3
8	70.4	100.0	60.2
24	70.4	100.0	93.6

Reaction 6: EtOAc



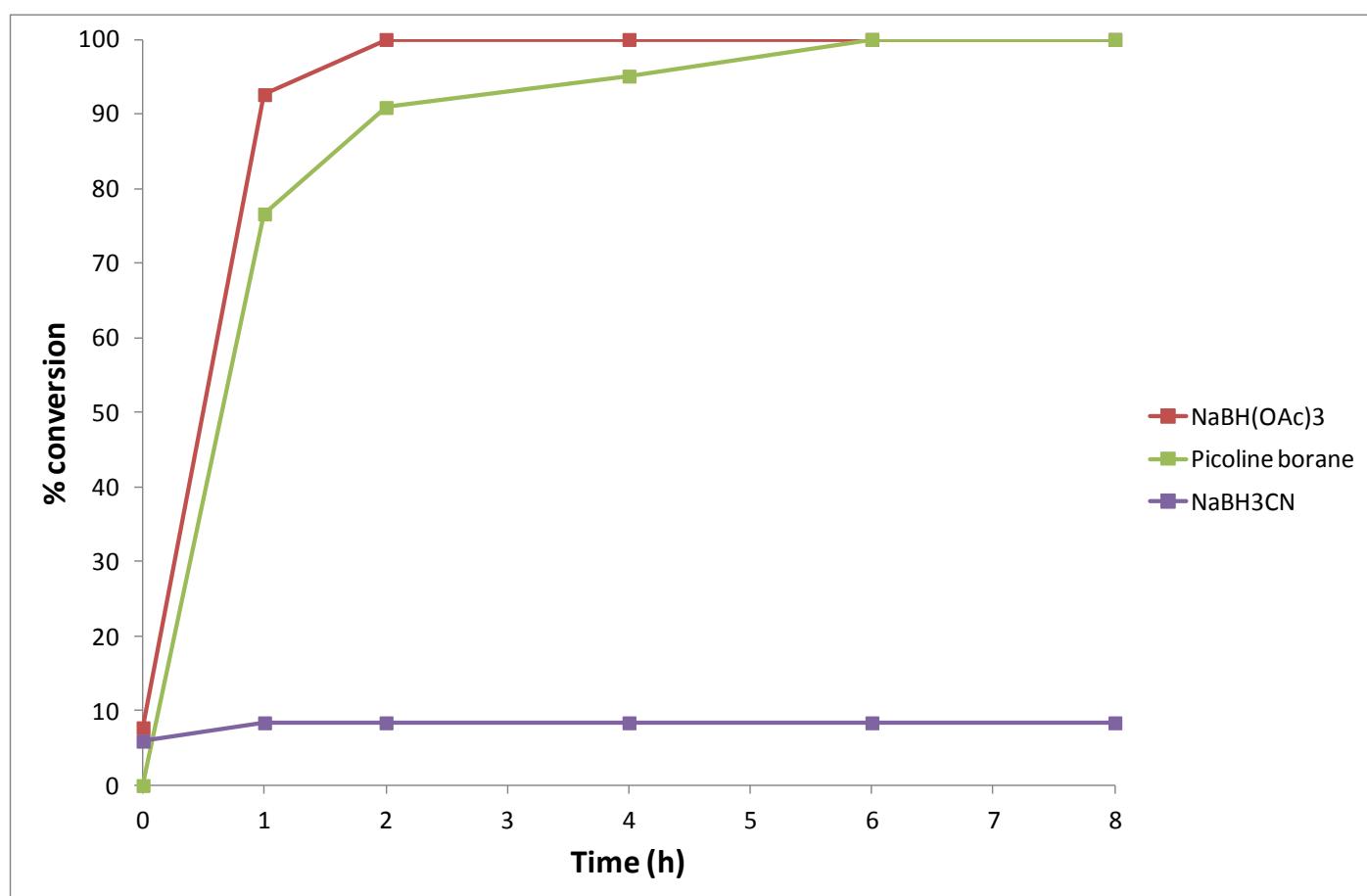
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	9.5	0.0	3.1
1	100.0	82.3	3.5
2	100.0	90.6	3.5
4	100.0	93.5	3.5
6	100.0	98.3	3.6
8	100.0	100.0	4.0
24	100.0	100.0	31.0

Reaction 6: IPA



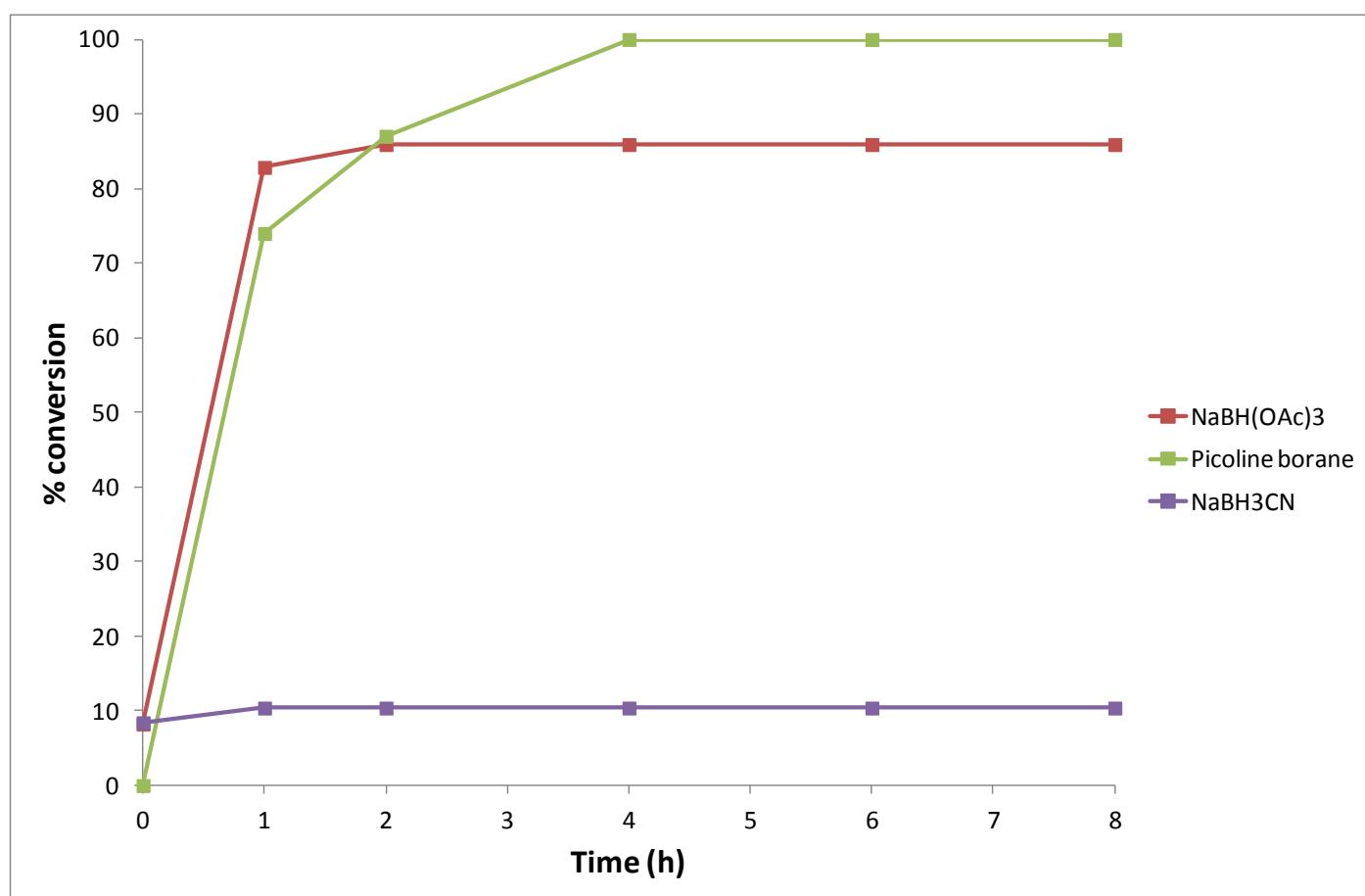
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	19.8	19.7	33.4
1	100.0	24.2	46.0
2	100.0	34.0	46.0
4	100.0	56.0	46.0
6	100.0	100.0	46.0
8	100.0	100.0	46.0
24	100.0	100.0	46.0

Reaction 6: 2-MeTHF



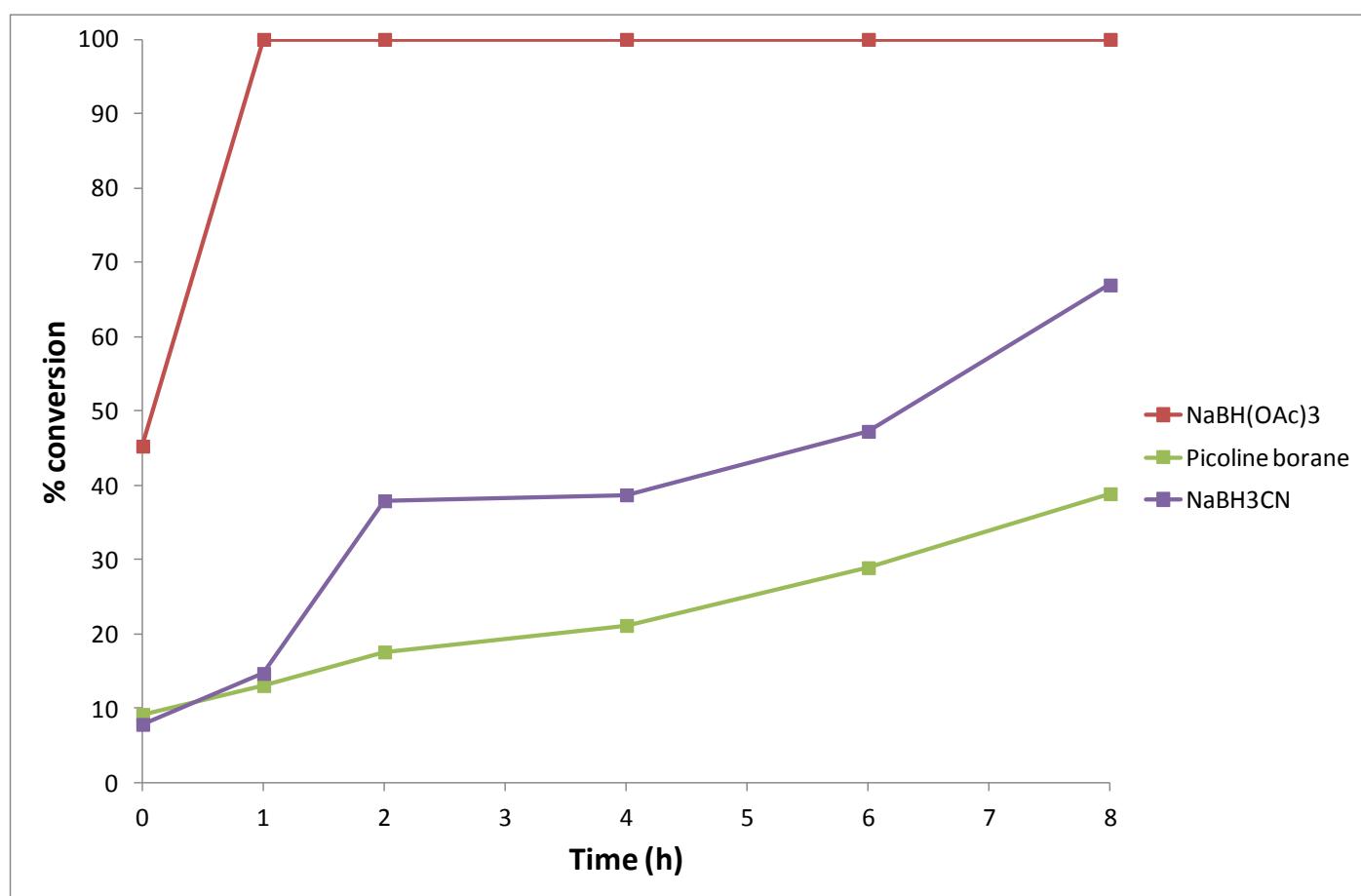
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	7.7	0.0	5.9
1	92.6	76.6	8.4
2	100.0	90.9	8.4
4	100.0	95.1	8.4
6	100.0	100.0	8.4
8	100.0	100.0	8.4
24	100.0	100.0	8.4

Reaction 6: THF



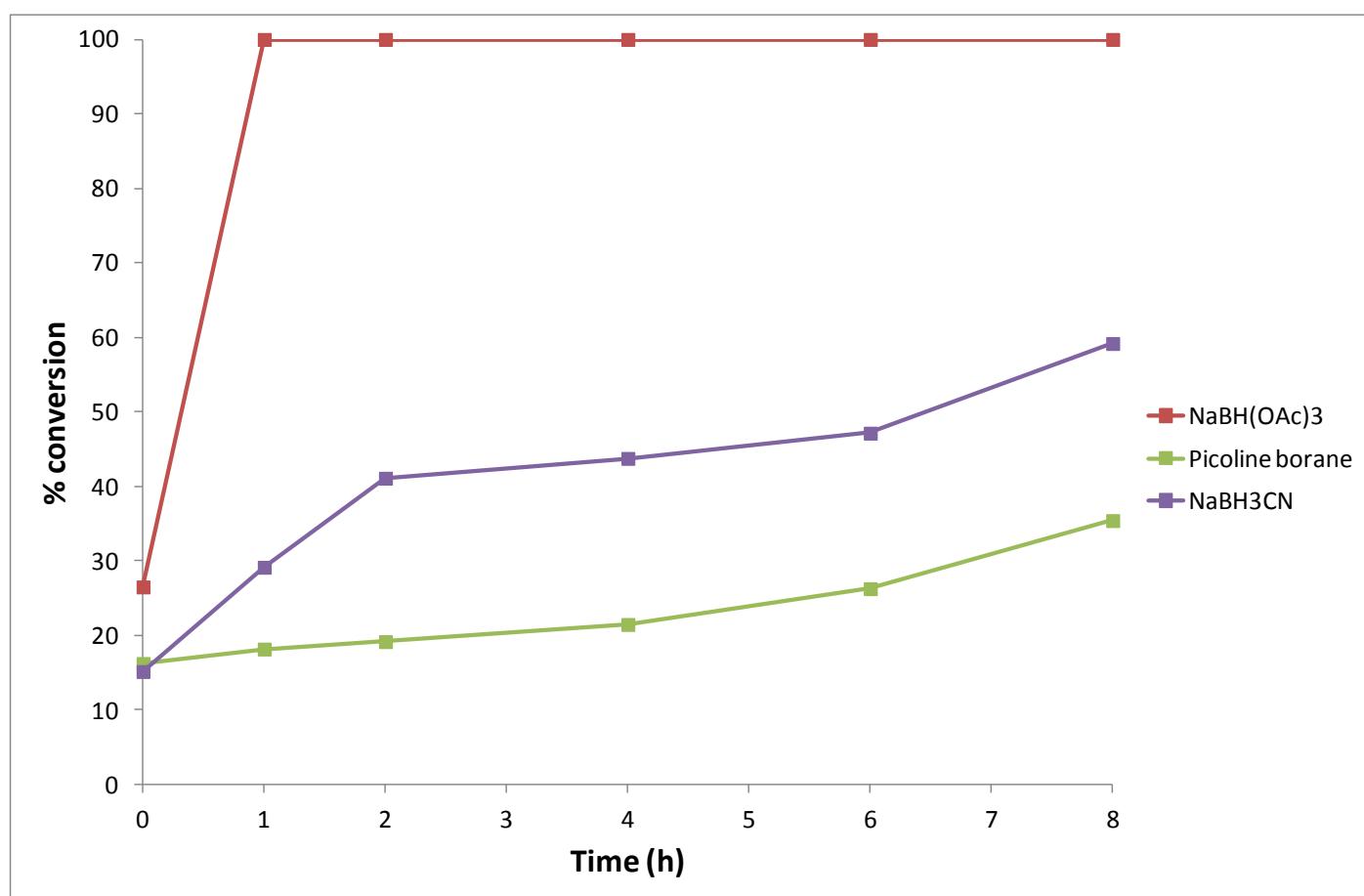
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	8.2	0.0	8.4
1	82.8	74.0	10.4
2	85.9	87.0	10.4
4	85.9	100.0	10.4
6	85.9	100.0	10.4
8	85.9	100.0	10.4
24	85.9	100.0	10.4

Reaction 7: TBME



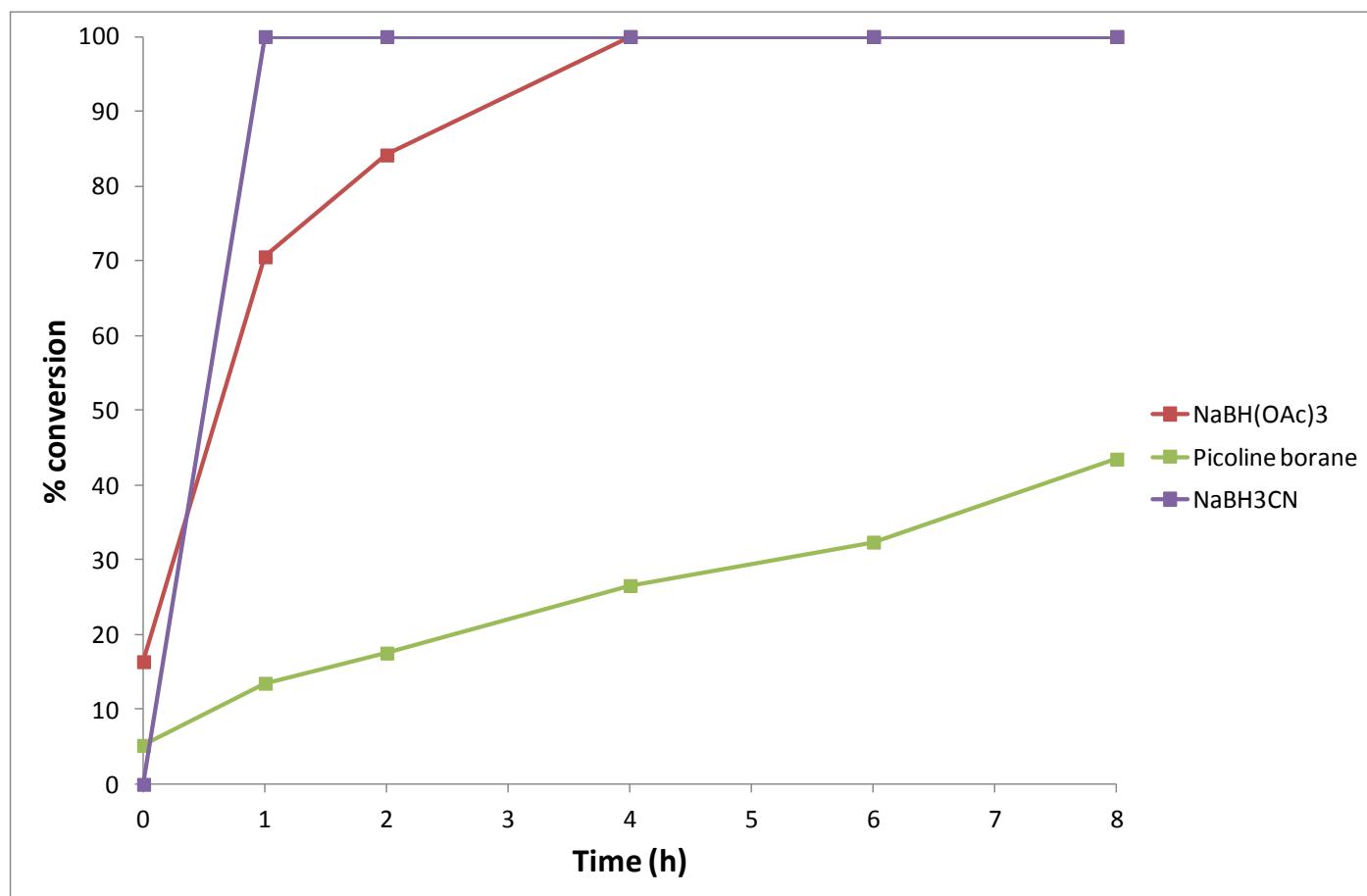
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	45.3	9.2	7.9
1	100.0	13.1	14.7
2	100.0	17.6	37.9
4	100.0	21.2	38.7
6	100.0	29.0	47.3
8	100.0	38.9	67.0
24	100.0	59.3	82.0

Reaction 7: CPME



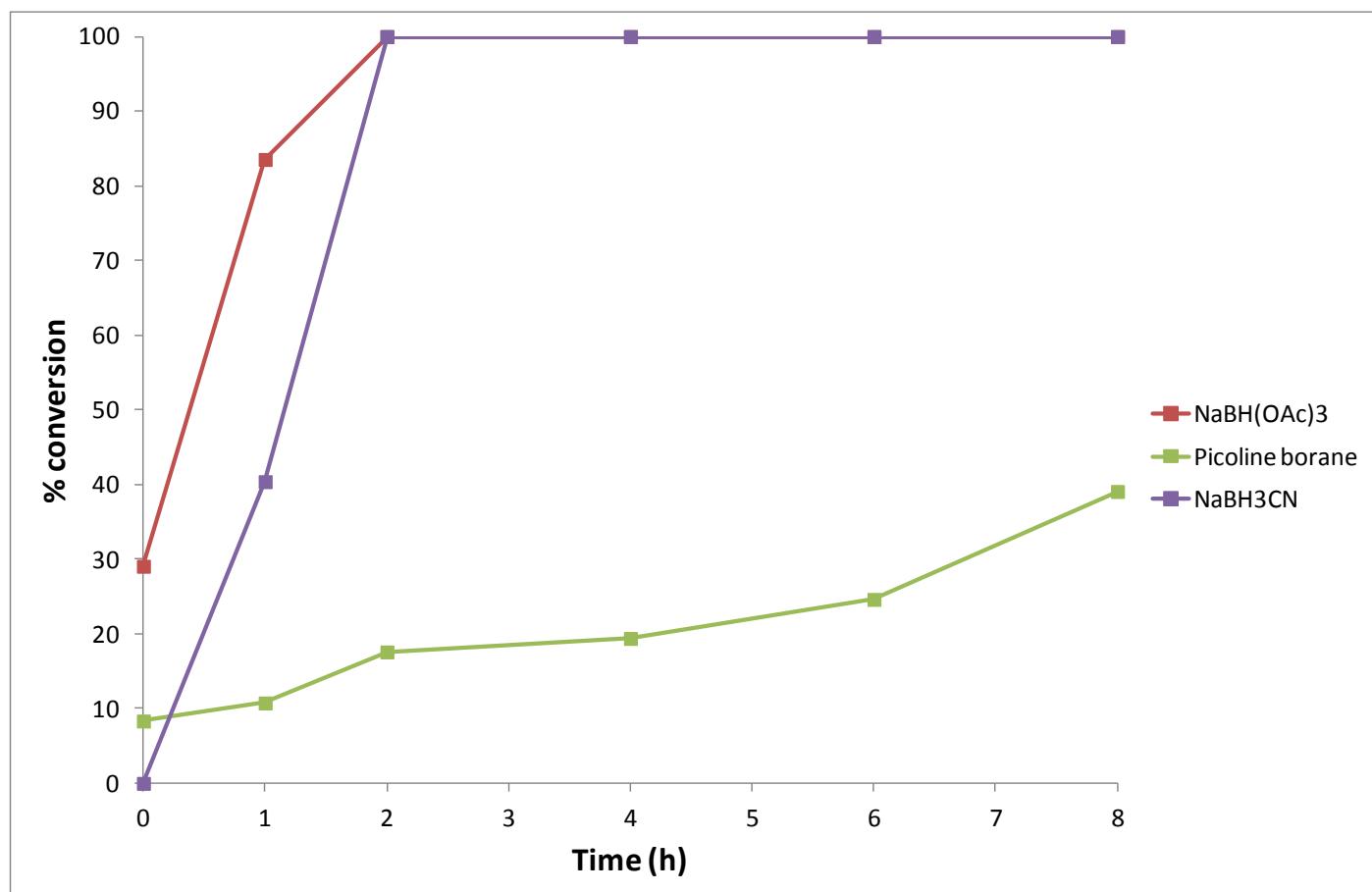
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	26.5	16.3	15.2
1	100.0	18.2	29.2
2	100.0	19.2	41.1
4	100.0	21.5	43.8
6	100.0	26.3	47.2
8	100.0	35.4	59.2
24	100.0	55.4	59.2

Reaction 7: DCE



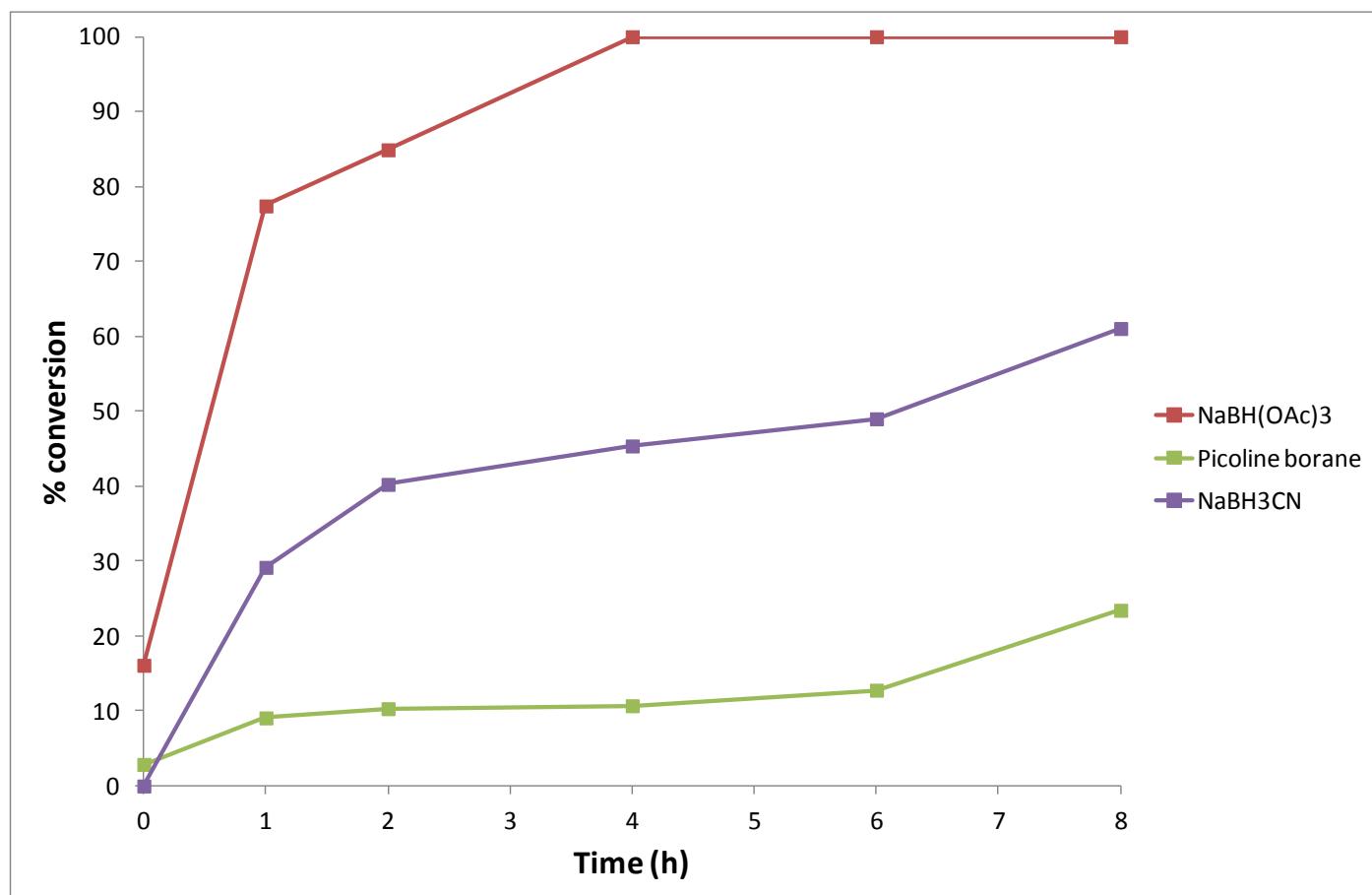
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	16.4	5.2	0.0
1	70.5	13.5	100.0
2	84.2	17.6	100.0
4	100.0	26.6	100.0
6	100.0	32.4	100.0
8	100.0	43.5	100.0
24	100.0	58.0	100.0

Reaction 7: CH₂Cl₂



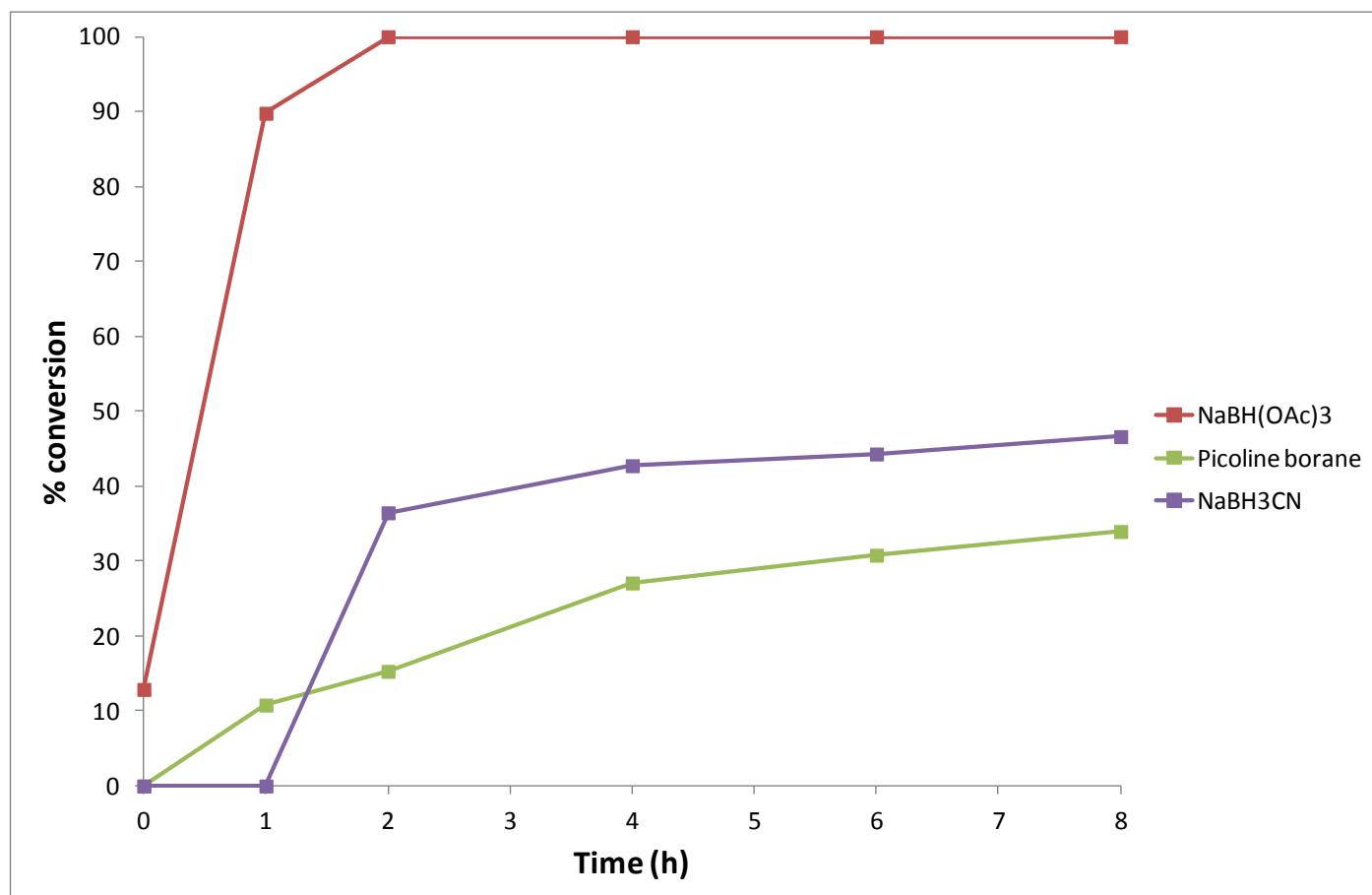
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	29.1	8.3	0.0
1	83.5	10.8	40.4
2	100.0	17.6	100.0
4	100.0	19.4	100.0
6	100.0	24.6	100.0
8	100.0	39.1	100.0
24	100.0	60.8	100.0

Reaction 7: DMC



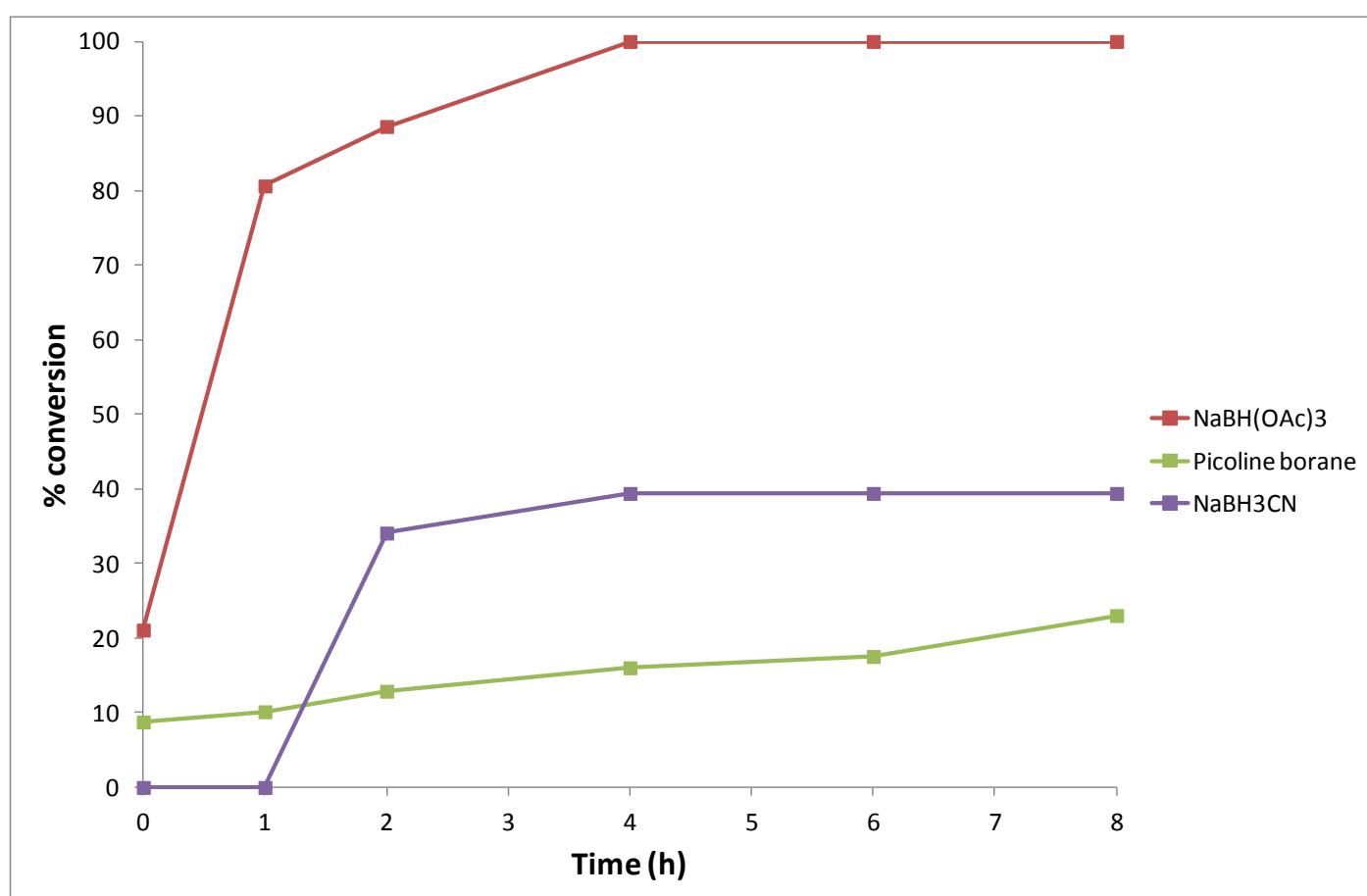
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	16.1	2.9	0.0
1	77.4	9.1	29.2
2	84.9	10.3	40.3
4	100.0	10.7	45.4
6	100.0	12.8	49.0
8	100.0	23.5	61.1
24	100.0	41.7	100.0

Reaction 7: DMF



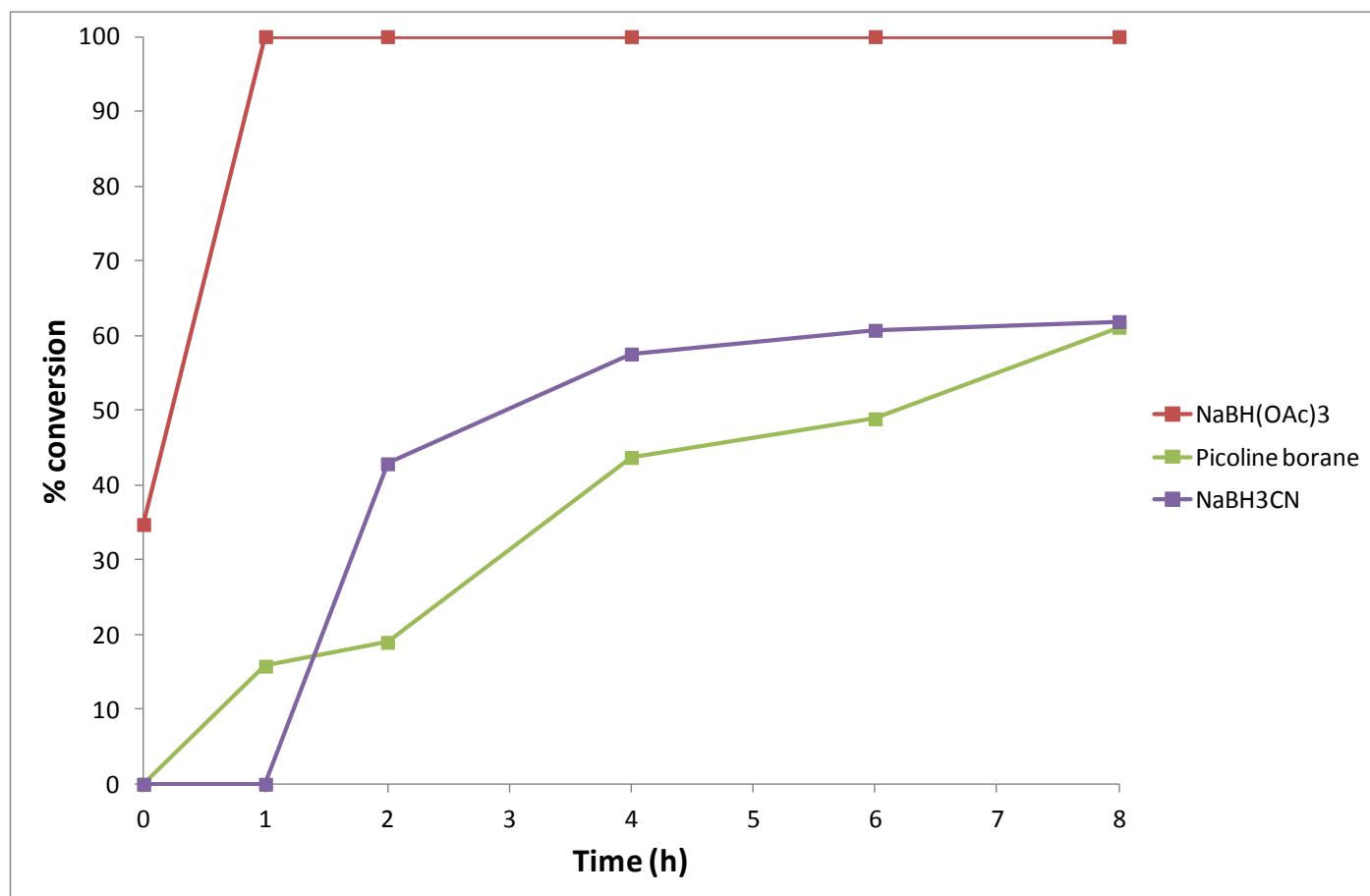
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	12.9	0.0	0.0
1	89.8	10.8	0.0
2	100.0	15.3	36.5
4	100.0	27.1	42.7
6	100.0	30.8	44.3
8	100.0	34.0	46.6
24	100.0	66.5	46.6

Reaction 7: EtOAc



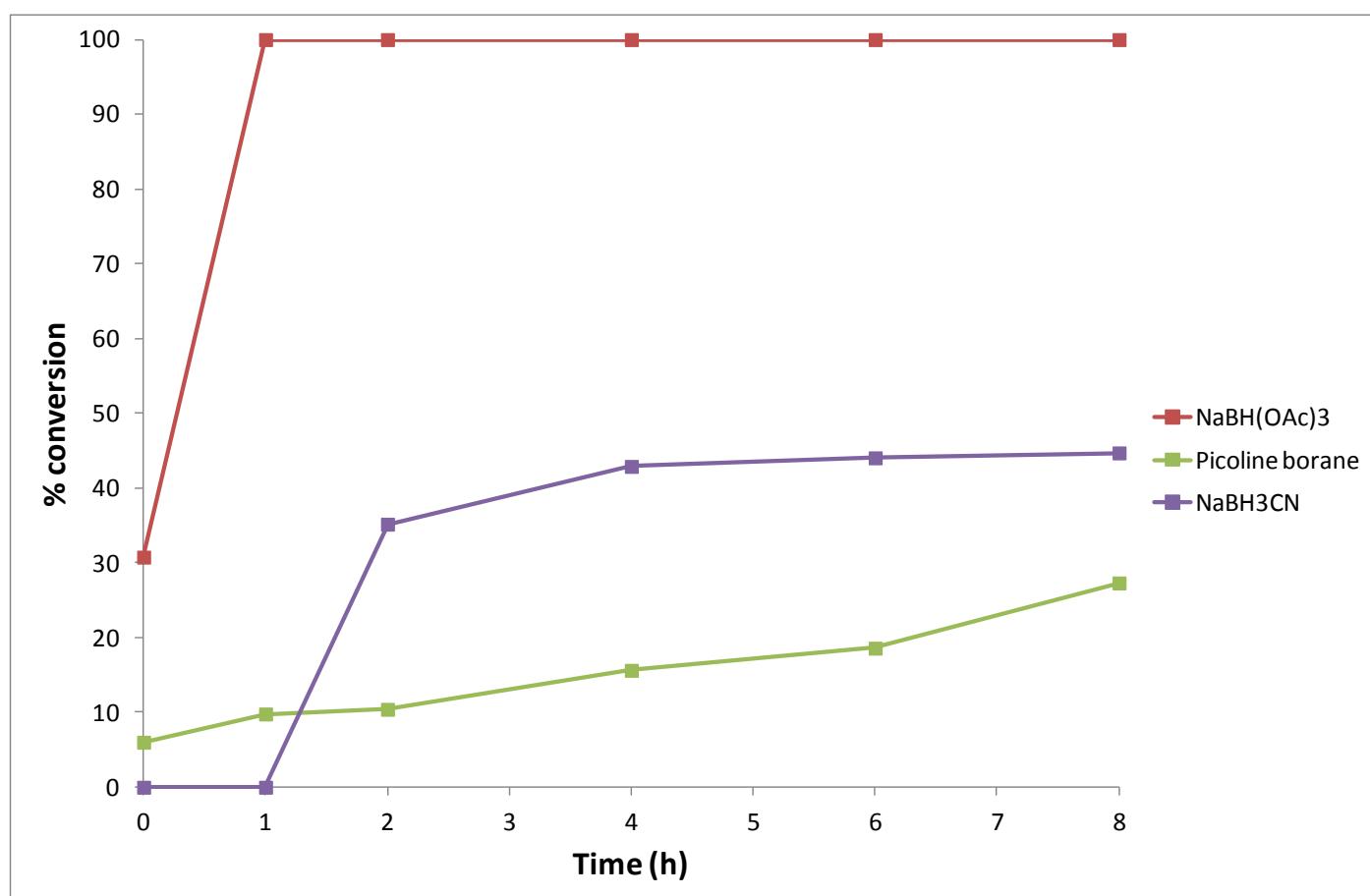
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	21.1	8.8	0.0
1	80.6	10.1	0.0
2	88.6	12.9	34.1
4	100.0	16.0	39.4
6	100.0	17.6	39.4
8	100.0	23.0	39.4
24	100.0	46.2	39.4

Reaction 7: IPA



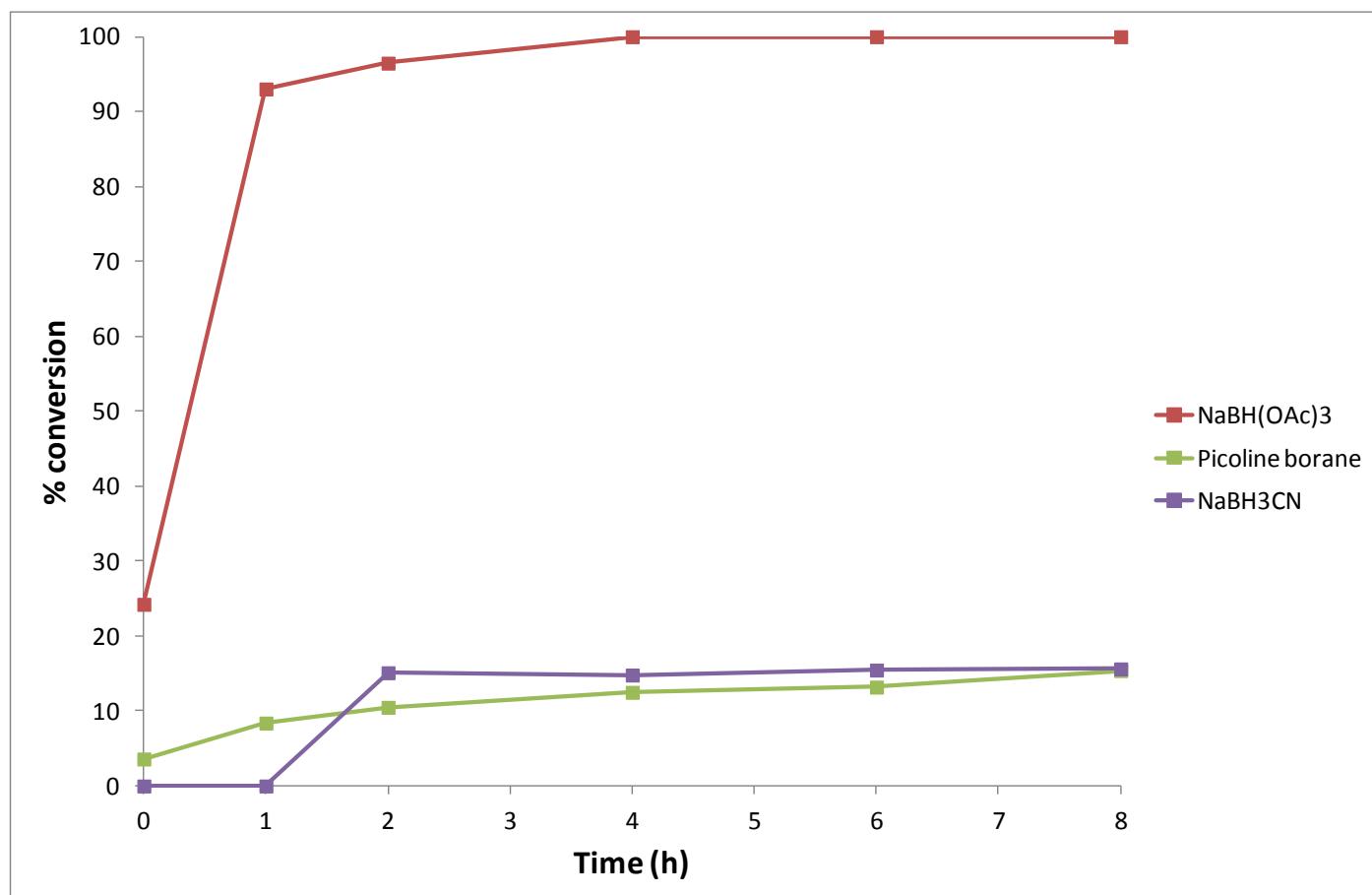
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	34.7	0.0	0.0
1	100.0	15.8	0.0
2	100.0	19.0	42.9
4	100.0	43.7	57.5
6	100.0	48.9	60.7
8	100.0	61.1	61.9
24	100.0	74.0	68.1

Reaction 7: 2-MeTHF



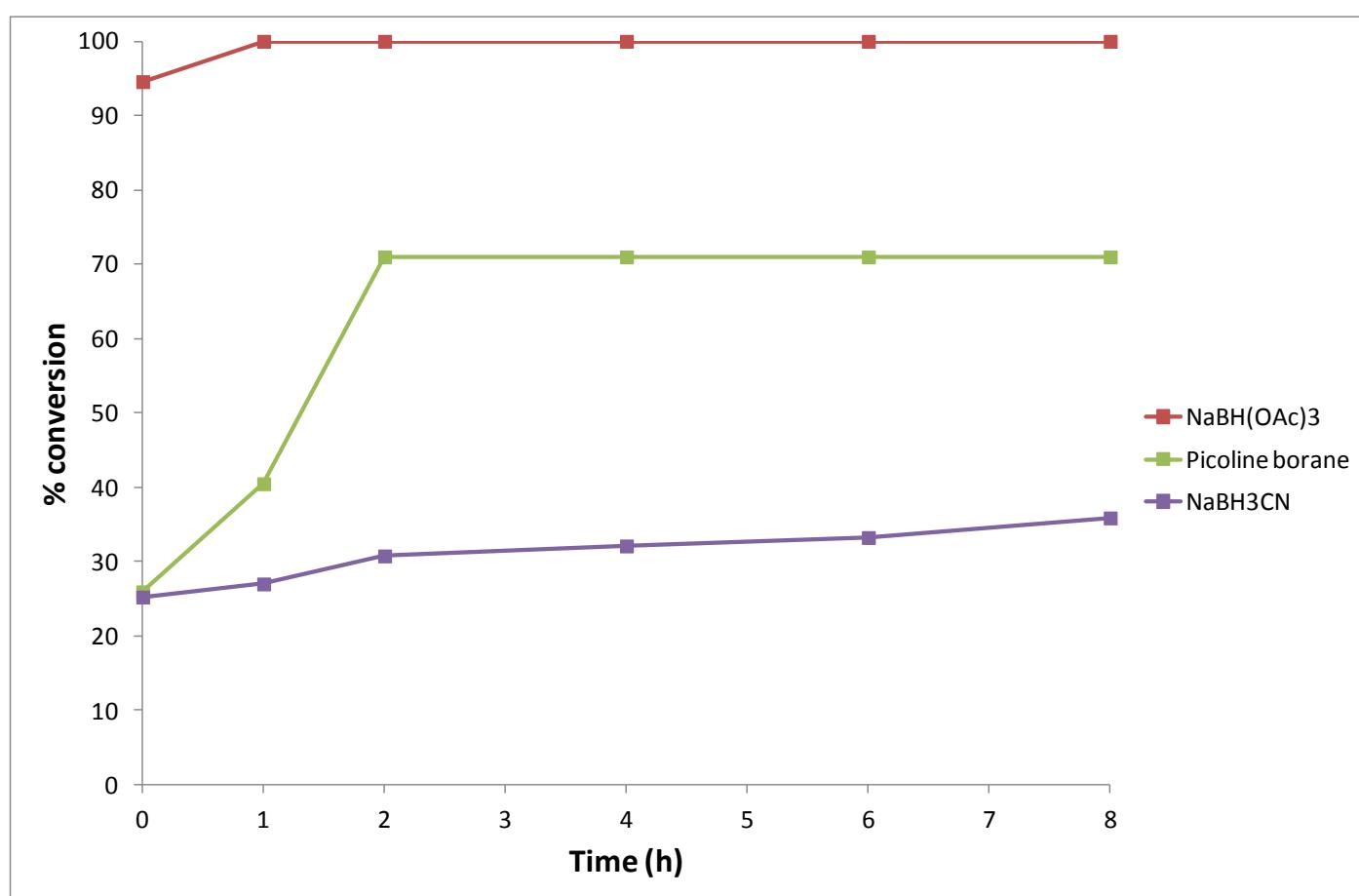
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	30.8	6.0	0.0
1	100.0	9.8	0.0
2	100.0	10.4	35.2
4	100.0	15.6	42.9
6	100.0	18.6	44.1
8	100.0	27.3	44.7
24	100.0	65.8	44.7

Reaction 7: THF



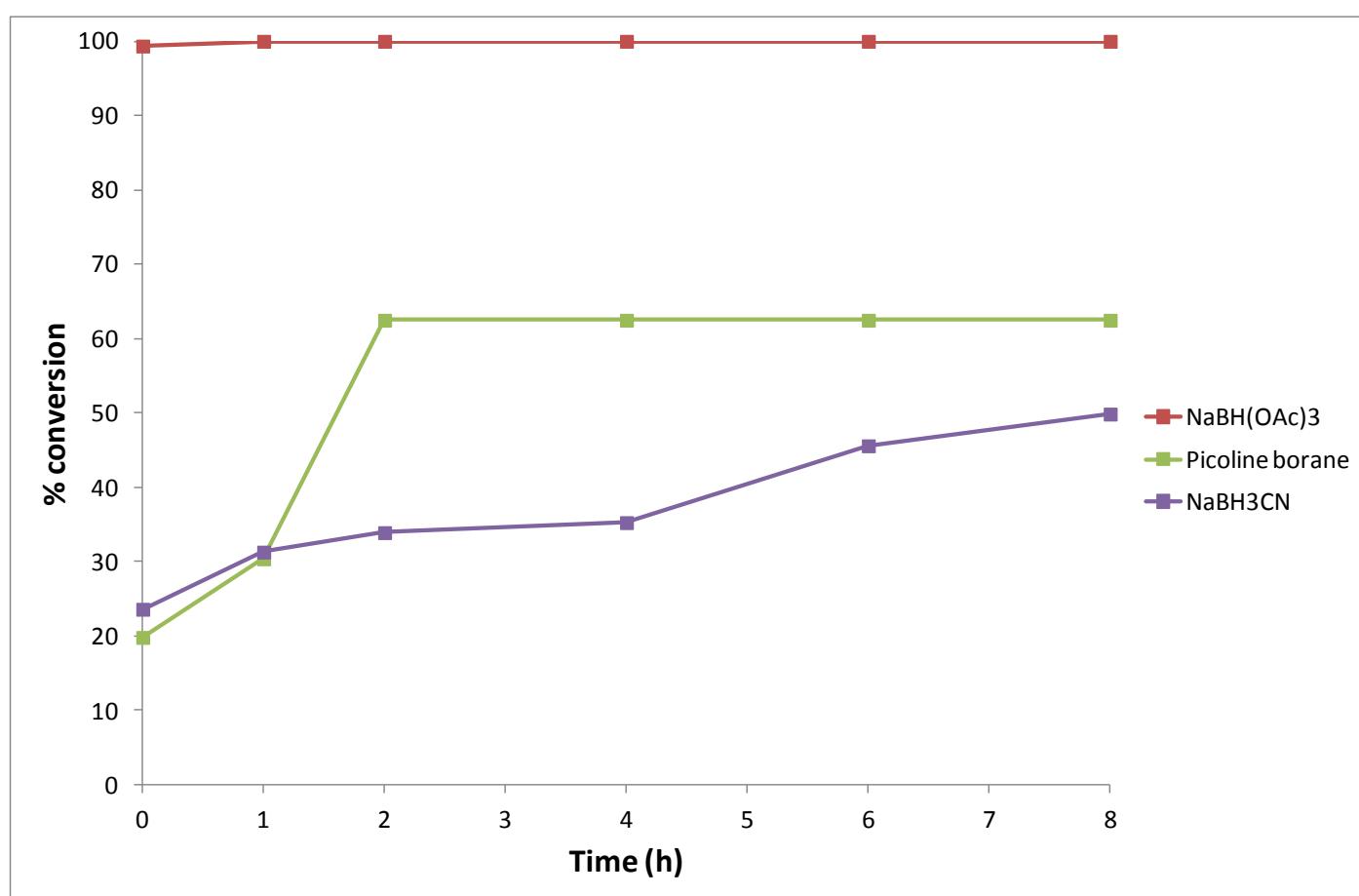
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	24.2	3.6	0.0
1	93.0	8.4	0.0
2	96.5	10.5	15.1
4	100.0	12.5	14.8
6	100.0	13.2	15.5
8	100.0	15.4	15.6
24	100.0	27.9	22.2

Reaction 8: TBME



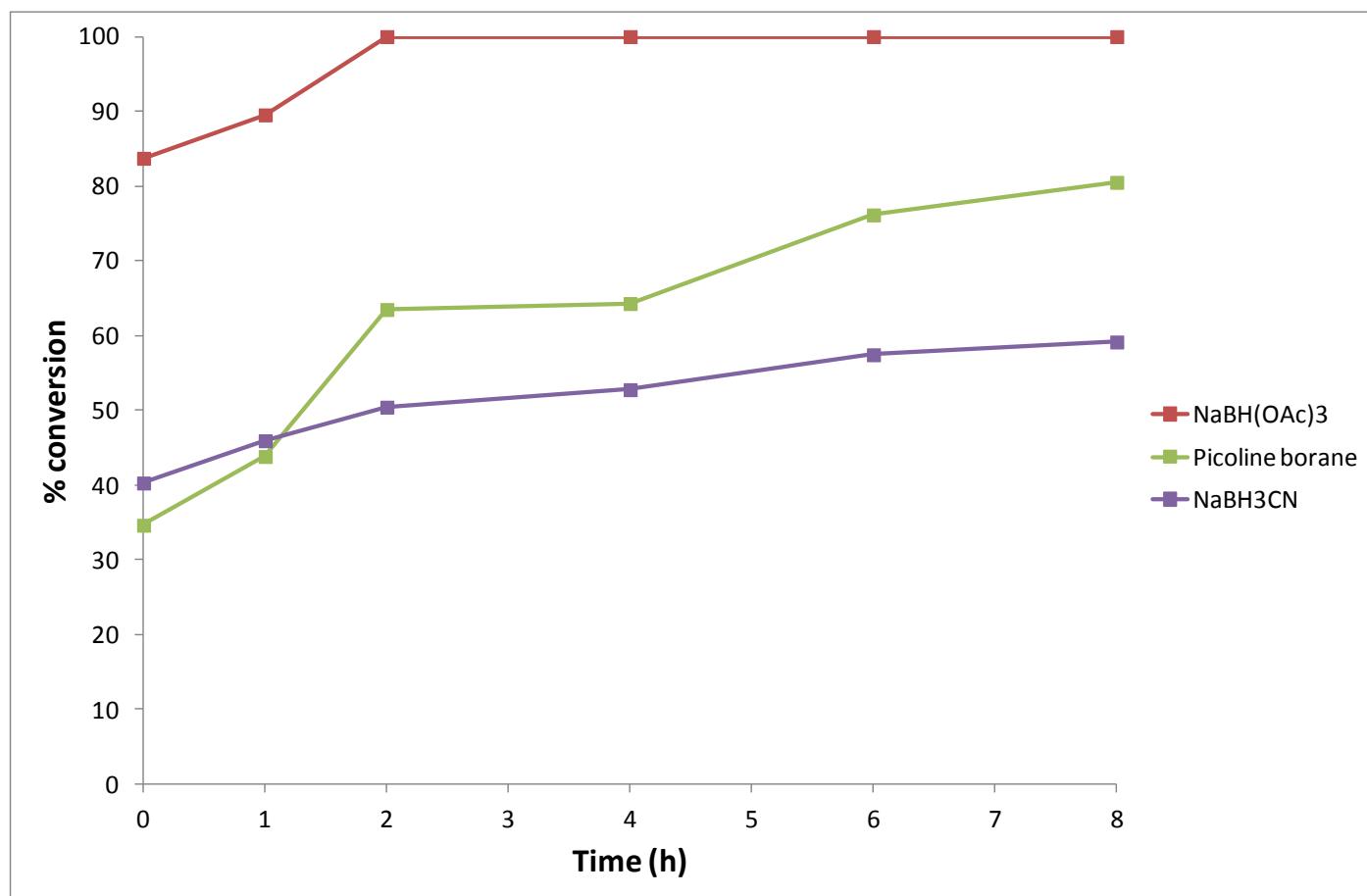
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	94.6	26.0	25.3
1	100.0	40.5	27.0
2	100.0	71.0	30.8
4	100.0	71.0	32.1
6	100.0	71.0	33.3
8	100.0	71.0	35.9
24	100.0	71.0	35.9

Reaction 8: CPME



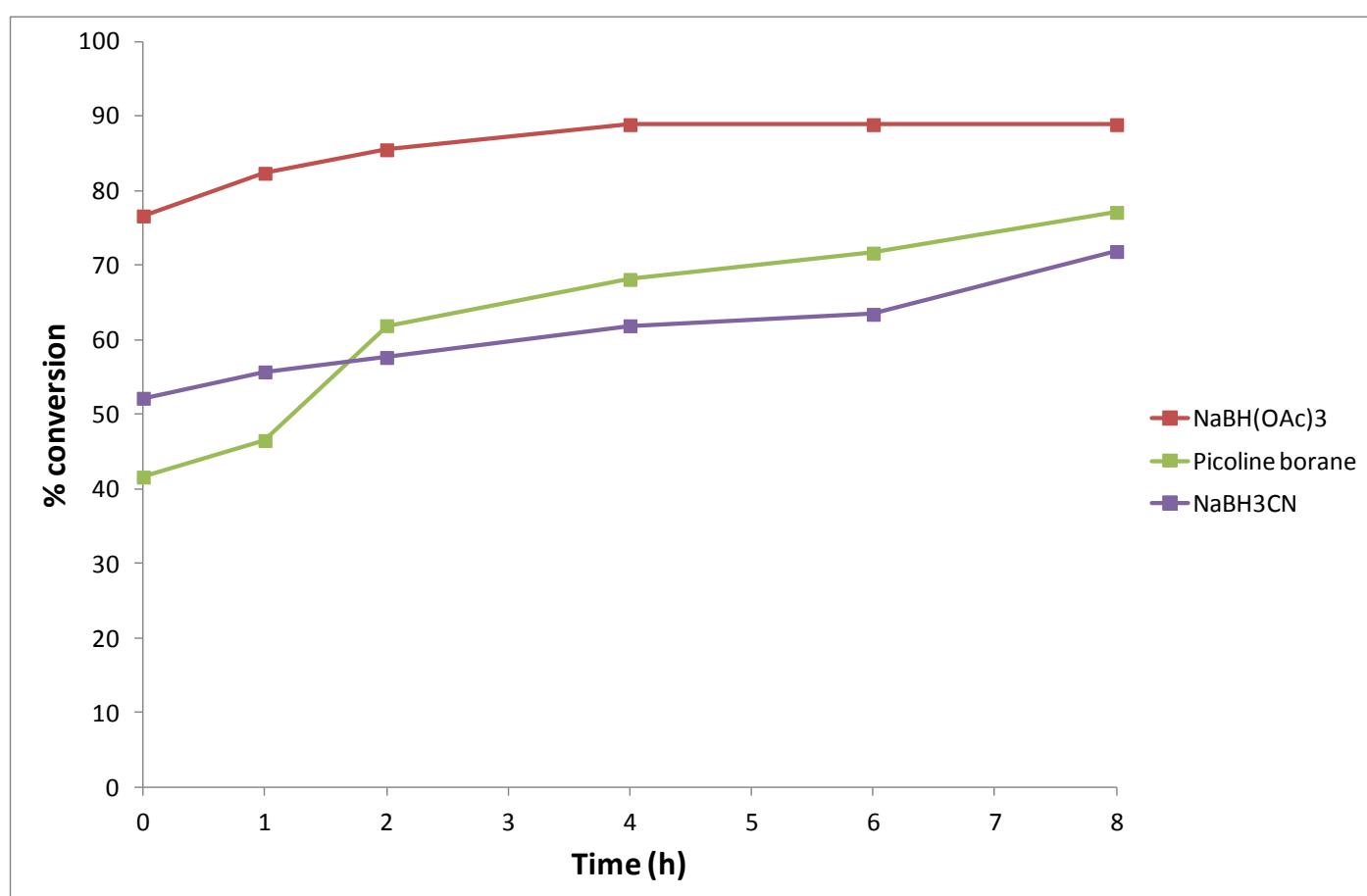
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	99.4	19.8	23.6
1	100.0	30.4	31.3
2	100.0	62.5	33.9
4	100.0	62.5	35.3
6	100.0	62.5	45.6
8	100.0	62.5	49.9
24	100.0	62.5	51.2

Reaction 8: DCE



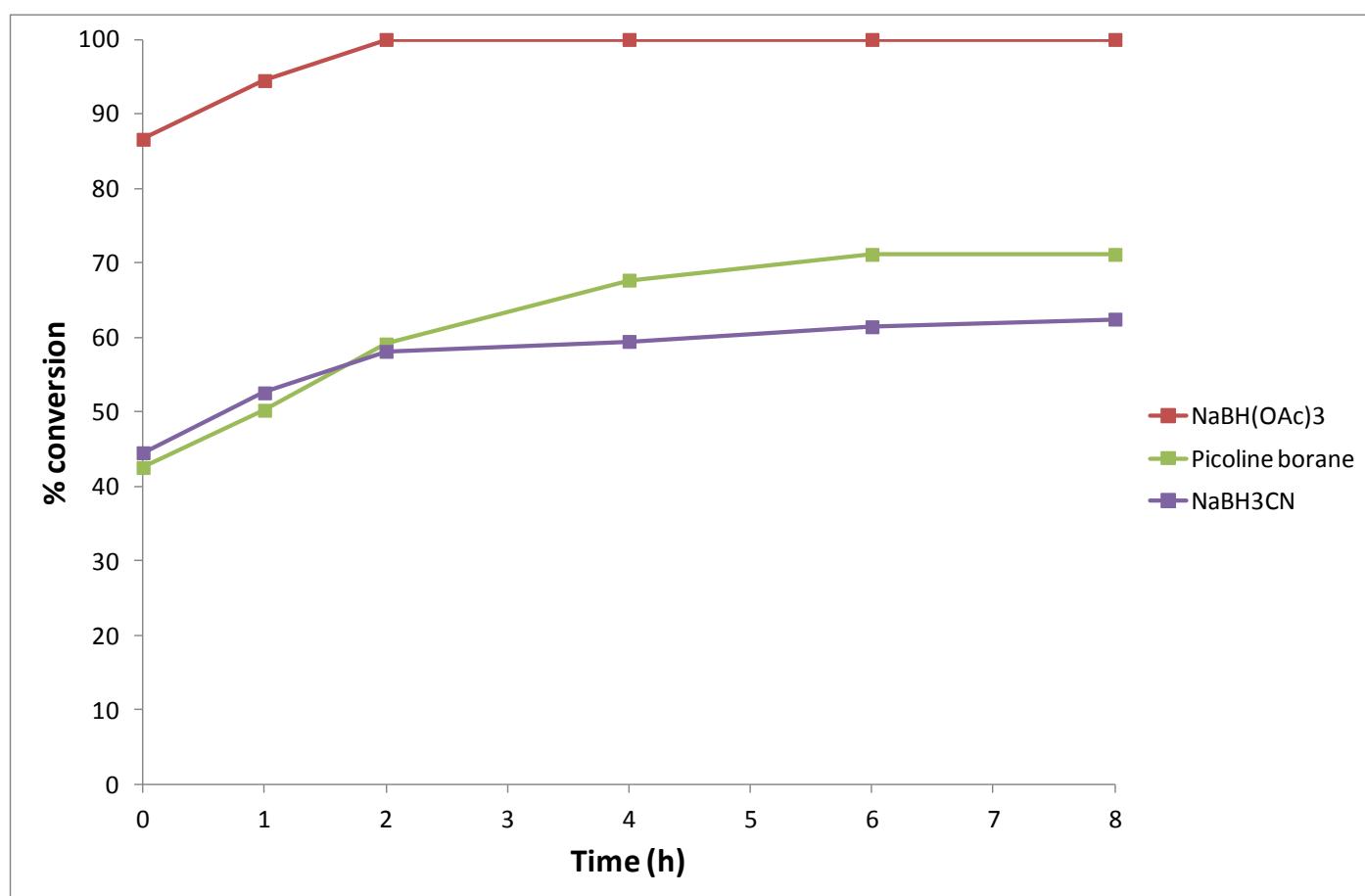
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	83.7	34.7	40.3
1	89.5	43.8	46.0
2	100.0	63.5	50.4
4	100.0	64.3	52.8
6	100.0	76.1	57.4
8	100.0	80.5	59.2
24	100.0	81.4	59.2

Reaction 8: CH₂Cl₂



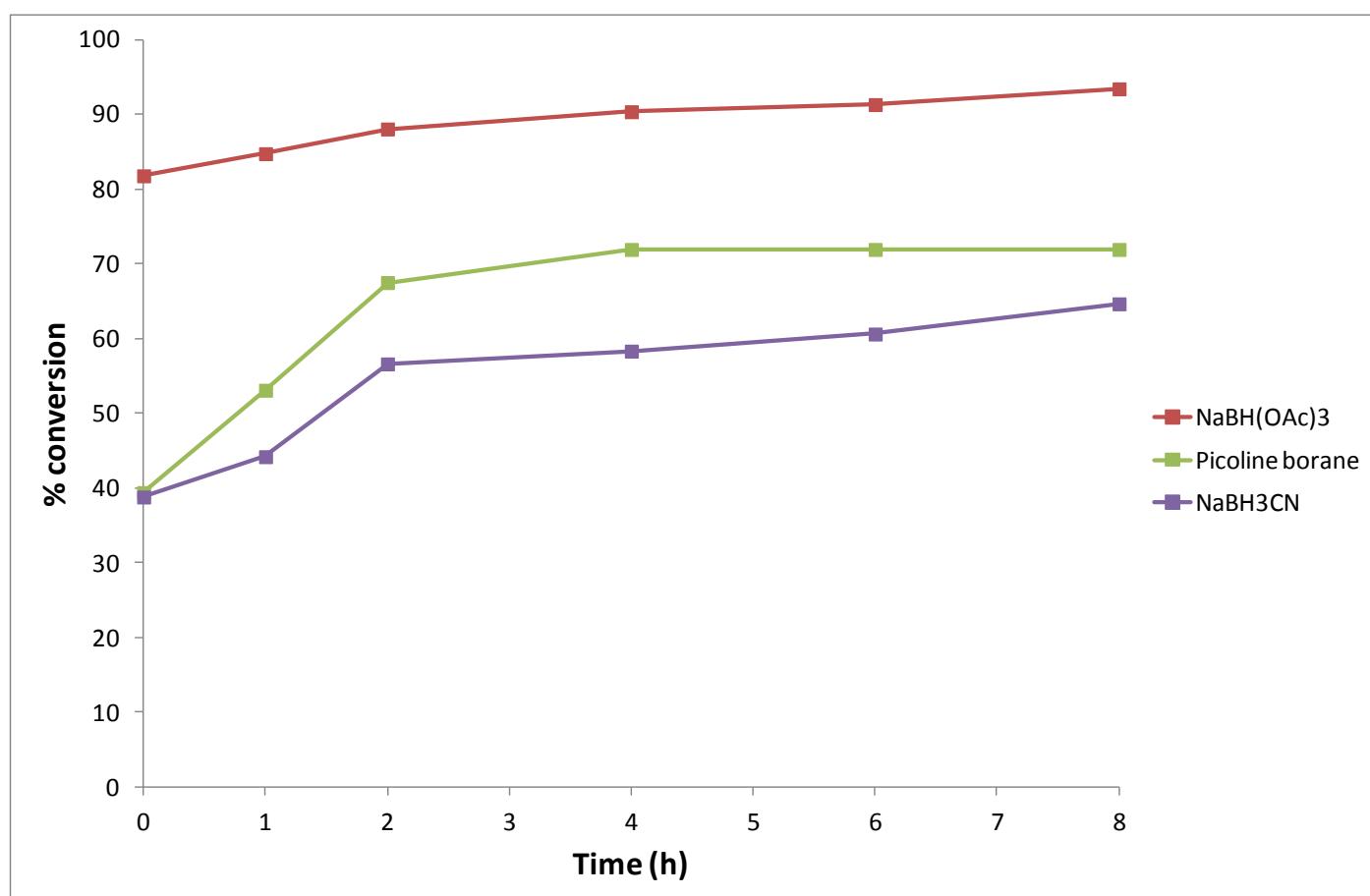
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	76.6	41.6	52.2
1	82.3	46.5	55.7
2	85.5	61.9	57.6
4	88.8	68.1	61.9
6	88.8	71.6	63.4
8	88.8	77.1	71.8
24	88.8	82.4	72.4

Reaction 8: DMC



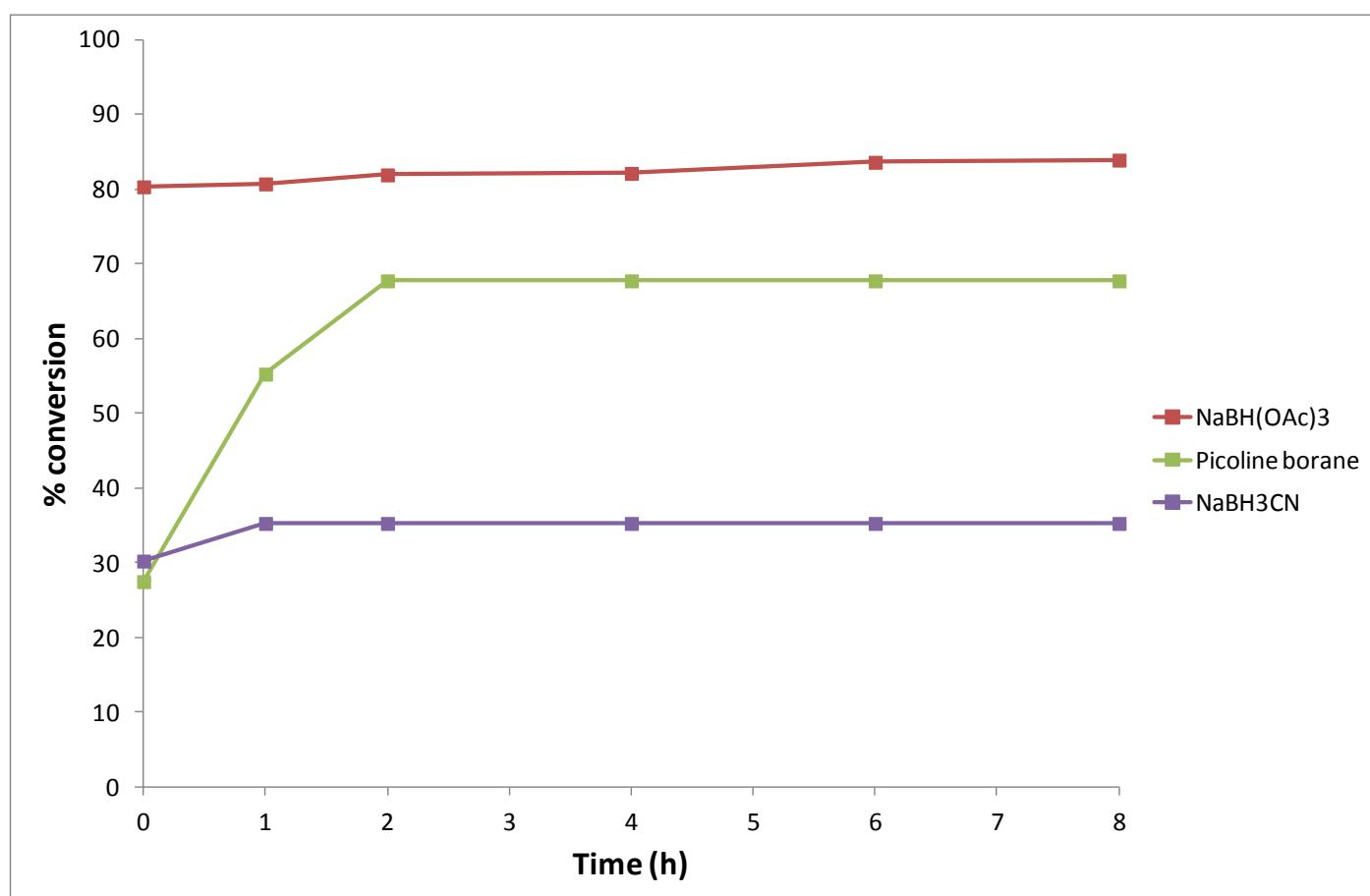
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	86.6	42.6	44.5
1	94.5	50.2	52.6
2	100.0	59.1	58.1
4	100.0	67.7	59.5
6	100.0	71.2	61.4
8	100.0	71.2	62.5
24	100.0	71.2	62.5

Reaction 8: DMF



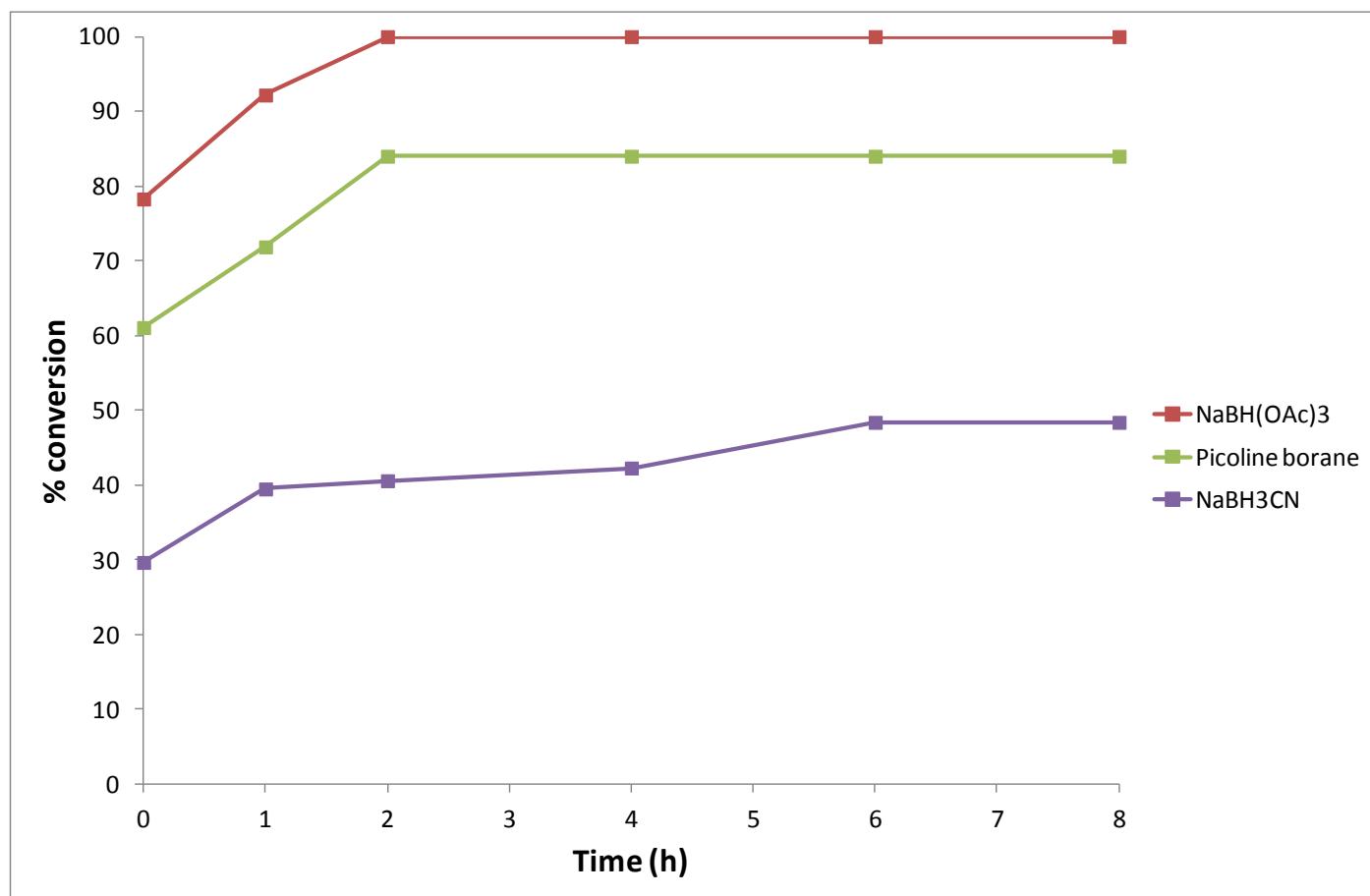
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	81.8	39.4	38.8
1	84.7	53.1	44.2
2	88.0	67.5	56.6
4	90.4	71.9	58.3
6	91.3	71.9	60.6
8	93.4	71.9	64.7
24	100.0	71.9	64.7

Reaction 8: EtOAc



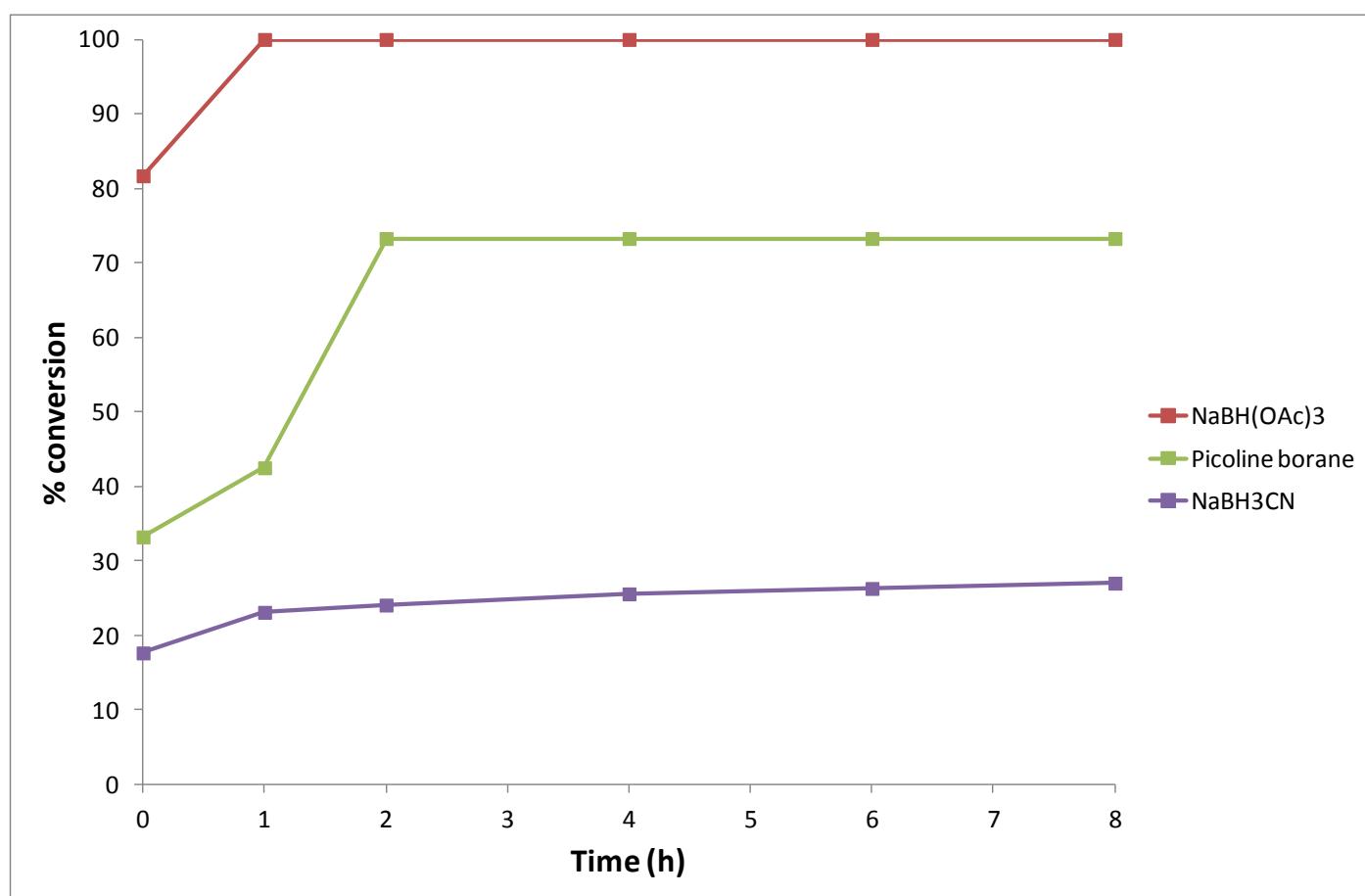
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	80.3	27.5	30.2
1	80.7	55.3	35.3
2	81.9	67.7	35.3
4	82.1	67.7	35.3
6	83.6	67.7	35.3
8	83.9	67.7	35.3
24	93.7	67.7	35.3

Reaction 8: IPA



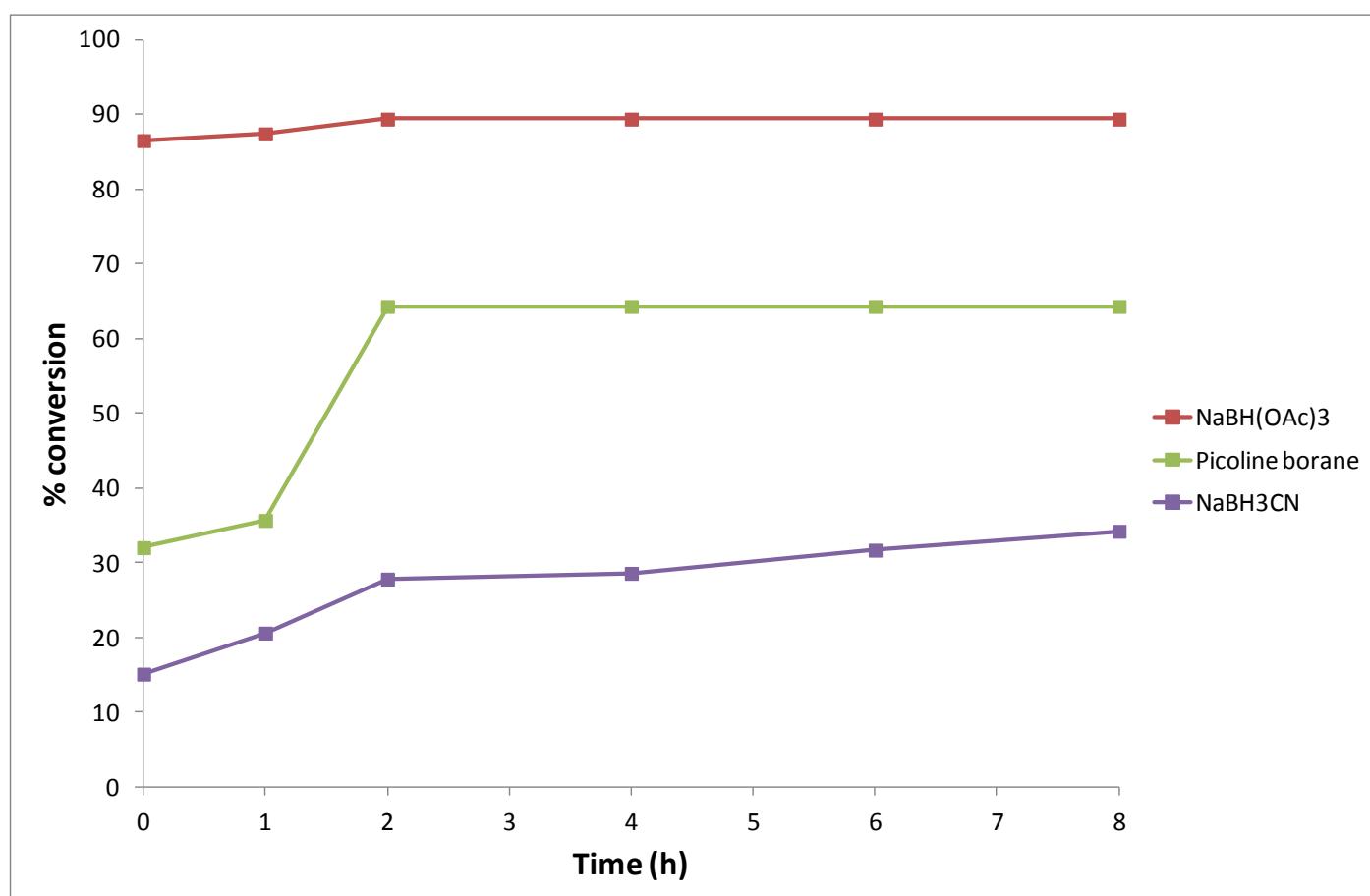
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	78.3	61.1	29.7
1	92.2	71.9	39.5
2	100.0	84.0	40.6
4	100.0	84.0	42.3
6	100.0	84.0	48.4
8	100.0	84.0	48.4
24	100.0	84.0	48.4

Reaction 8: 2-MeTHF



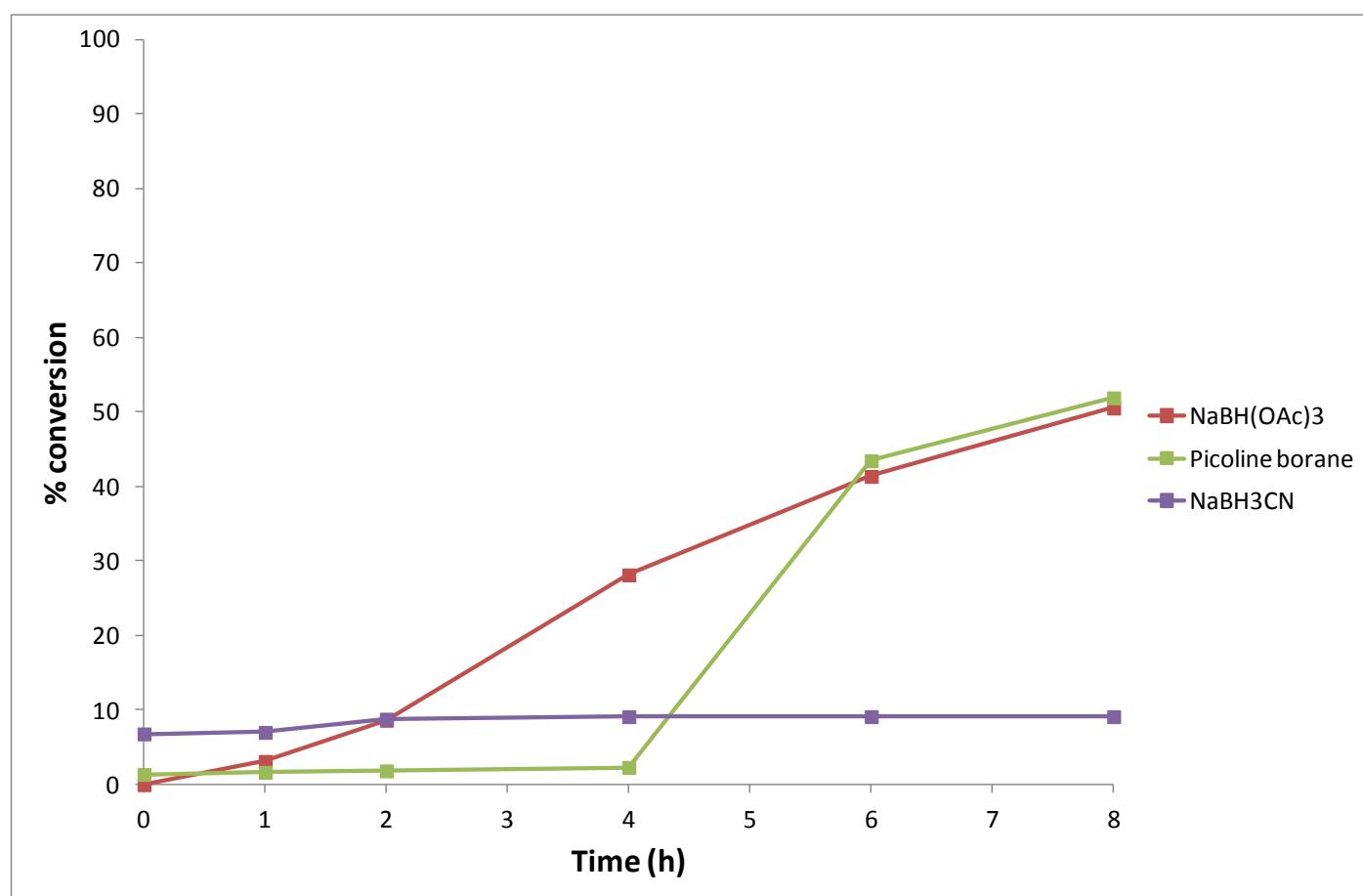
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	81.7	33.2	17.7
1	100.0	42.5	23.1
2	100.0	73.3	24.1
4	100.0	73.3	25.6
6	100.0	73.3	26.3
8	100.0	73.3	27.0
24	100.0	73.3	27.0

Reaction 8: THF



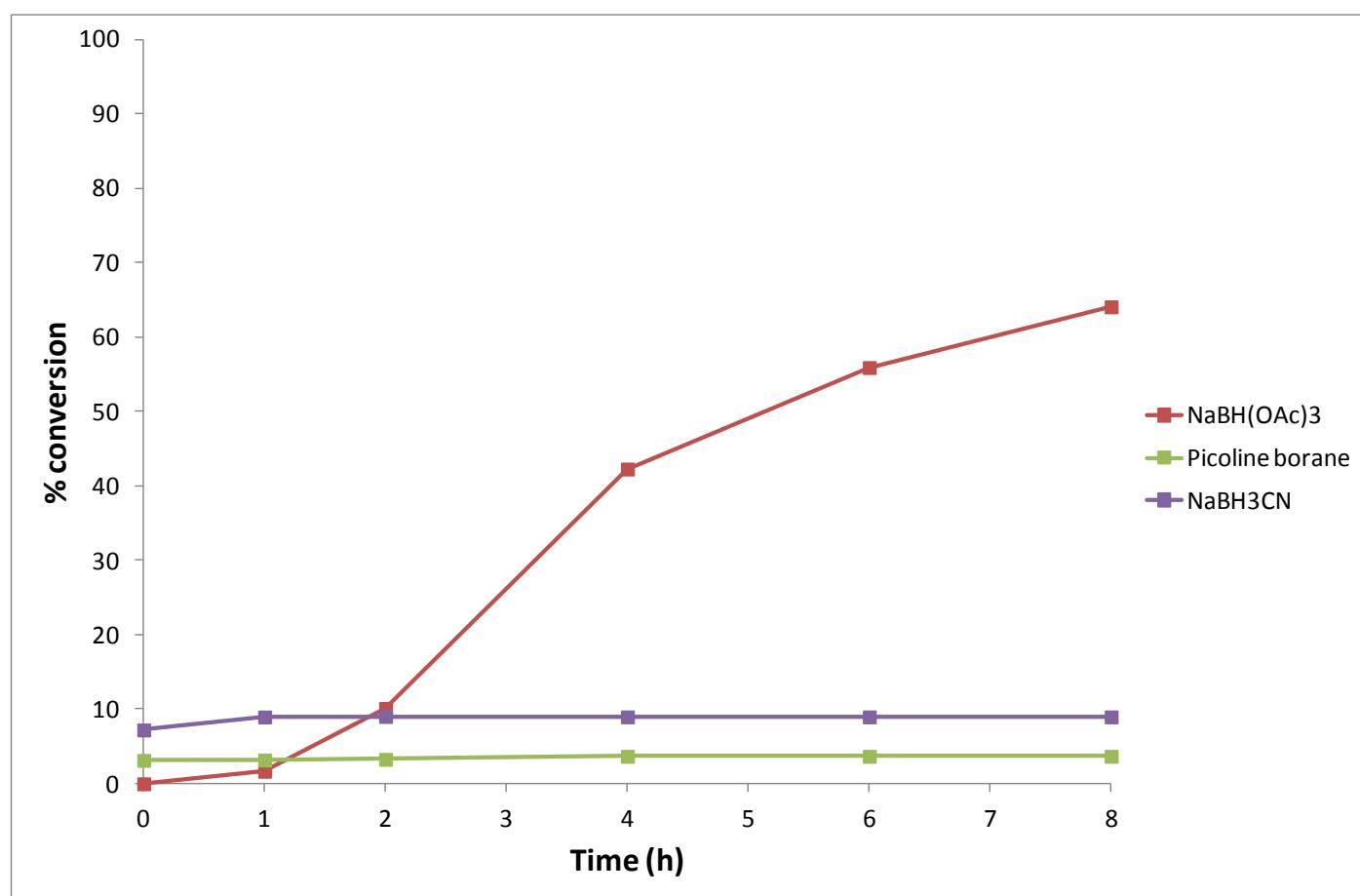
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	86.5	32.0	15.1
1	87.4	35.7	20.6
2	89.4	64.3	27.8
4	89.4	64.3	28.6
6	89.4	64.3	31.7
8	89.4	64.3	34.2
24	89.4	64.3	34.7

Reaction 9: TBME



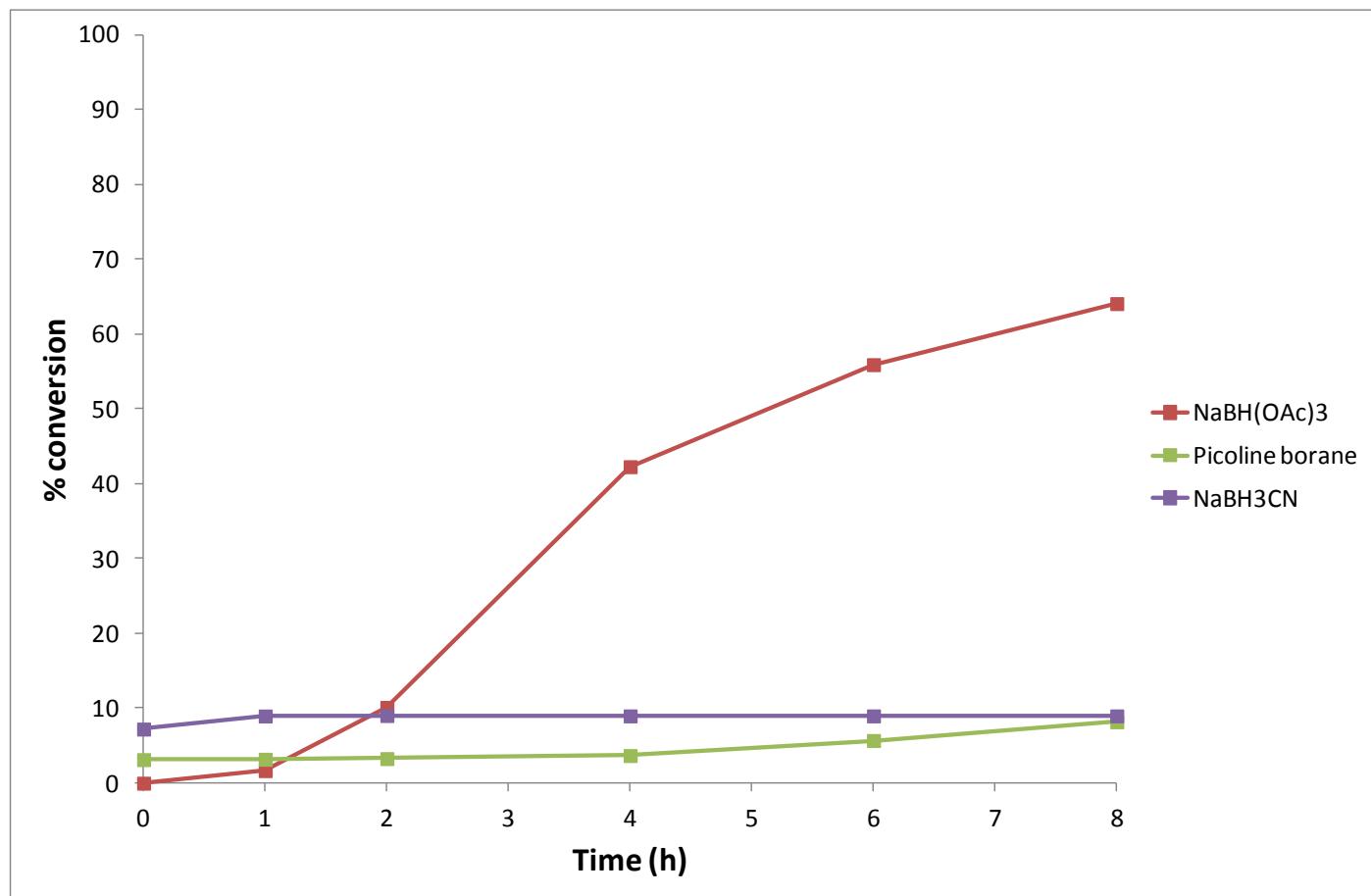
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	0.0	1.4	6.8
1	3.1	1.7	7.0
2	8.6	1.9	8.8
4	28.1	2.3	9.1
6	41.4	43.5	9.1
8	50.5	51.9	9.1
24	73.8	51.9	9.1

Reaction 9: CPME



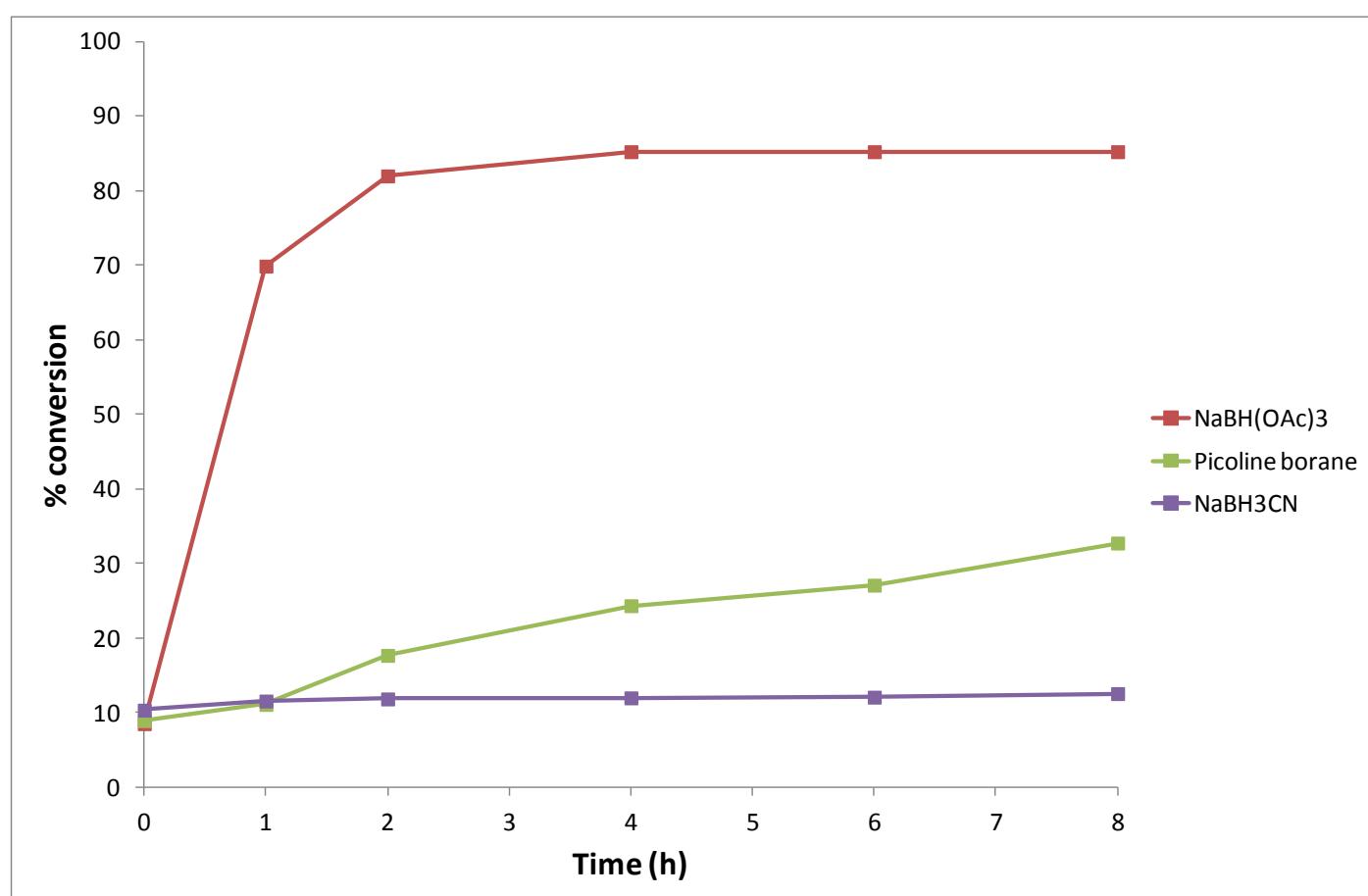
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	0.0	3.1	7.2
1	1.7	3.2	9.0
2	10.1	3.3	9.0
4	42.3	3.7	9.0
6	55.9	3.7	9.0
8	64.1	3.7	9.0
24	81.4	3.7	9.0

Reaction 9: DCE



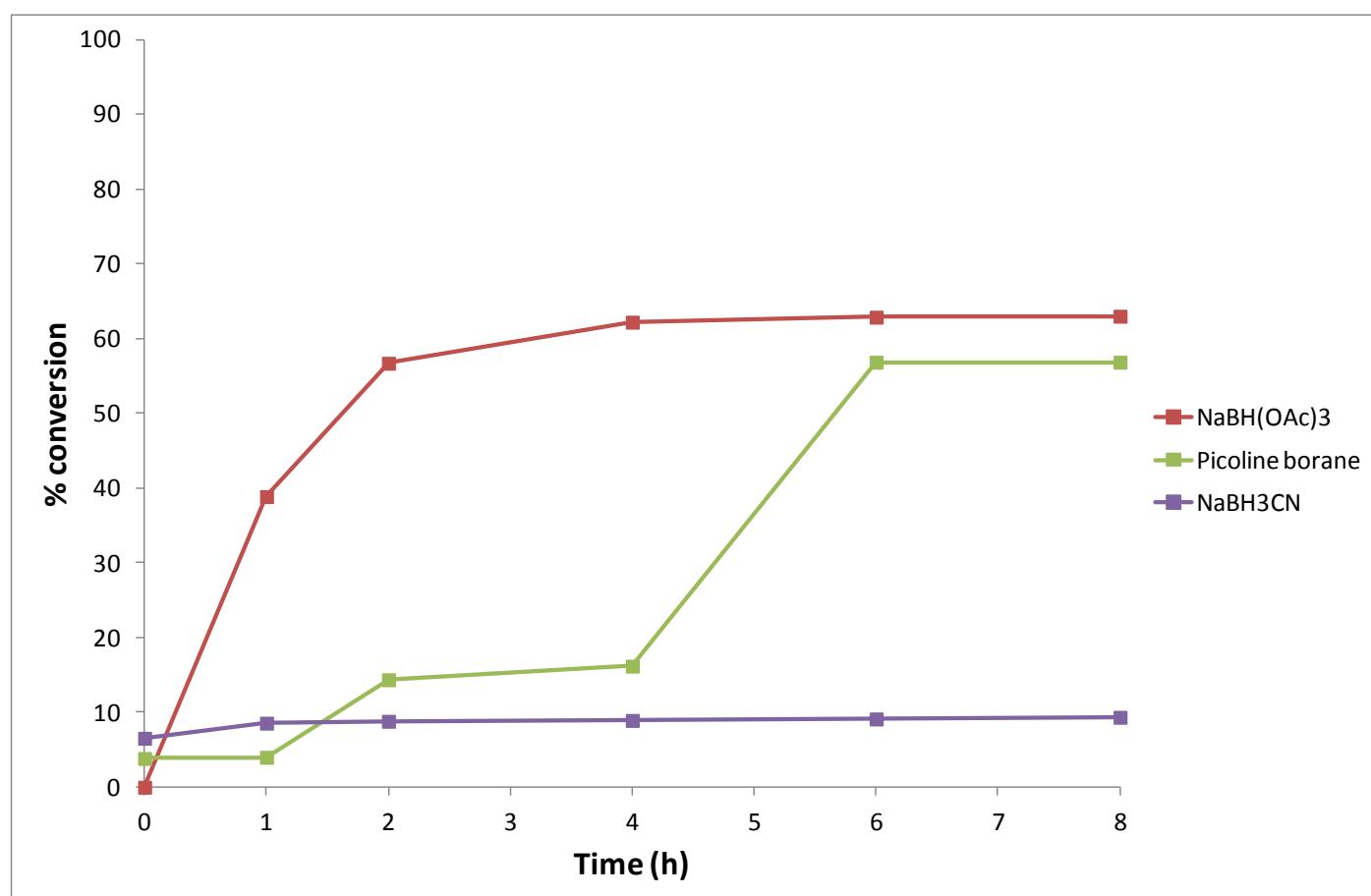
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	0.0	8.1	3.2
1	57.8	12.1	26.1
2	73.8	17.5	26.4
4	79.4	24.5	26.4
6	79.4	26.1	26.4
8	79.4	29.5	26.4
24	79.4	61.4	26.4

Reaction 9: CH₂Cl₂



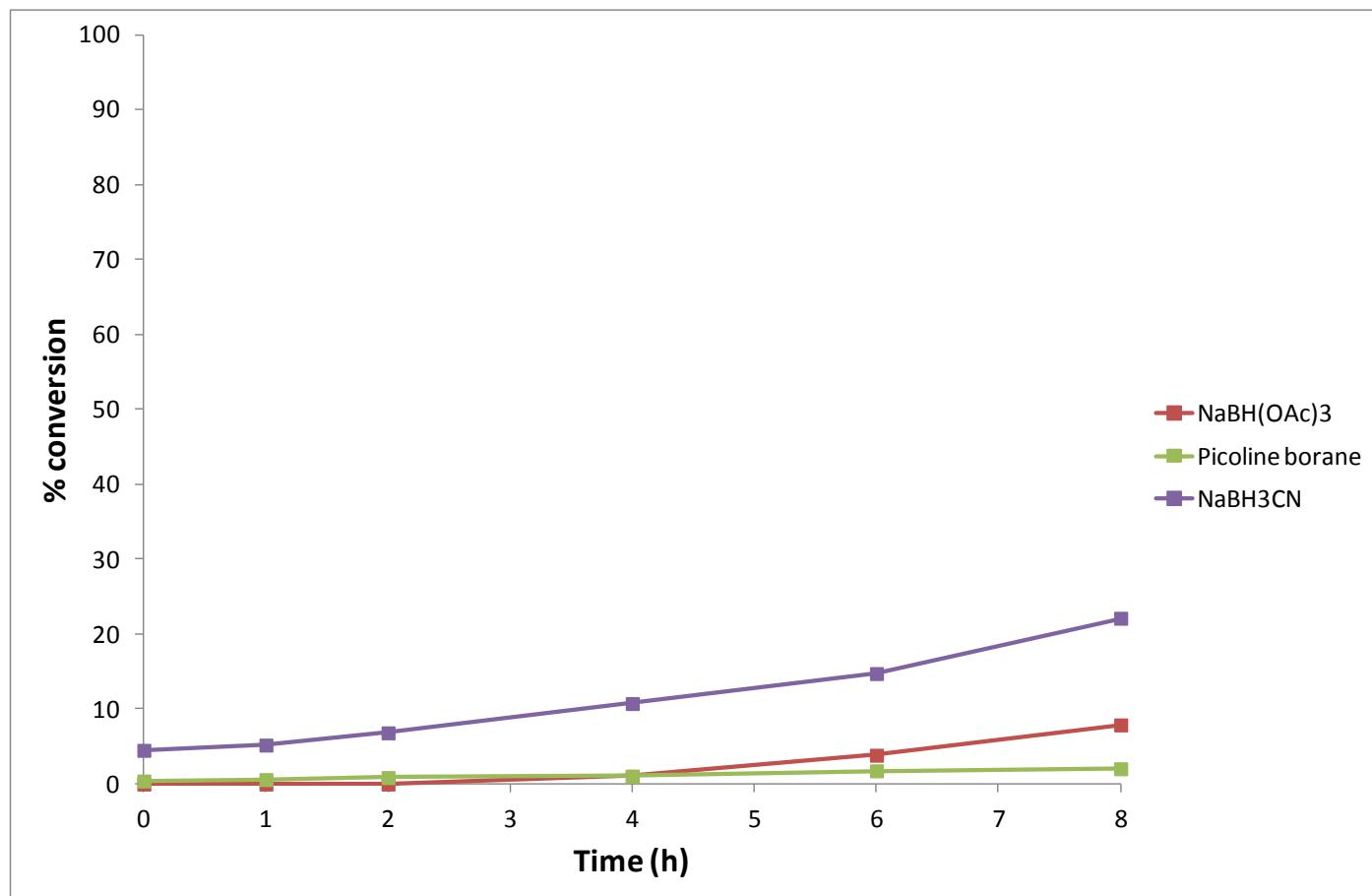
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	8.6	9.0	10.4
1	69.9	11.1	11.6
2	82.0	17.7	11.9
4	85.2	24.4	12.0
6	85.2	27.1	12.1
8	85.2	32.8	12.6
24	85.2	69.7	12.6

Reaction 9: DMC



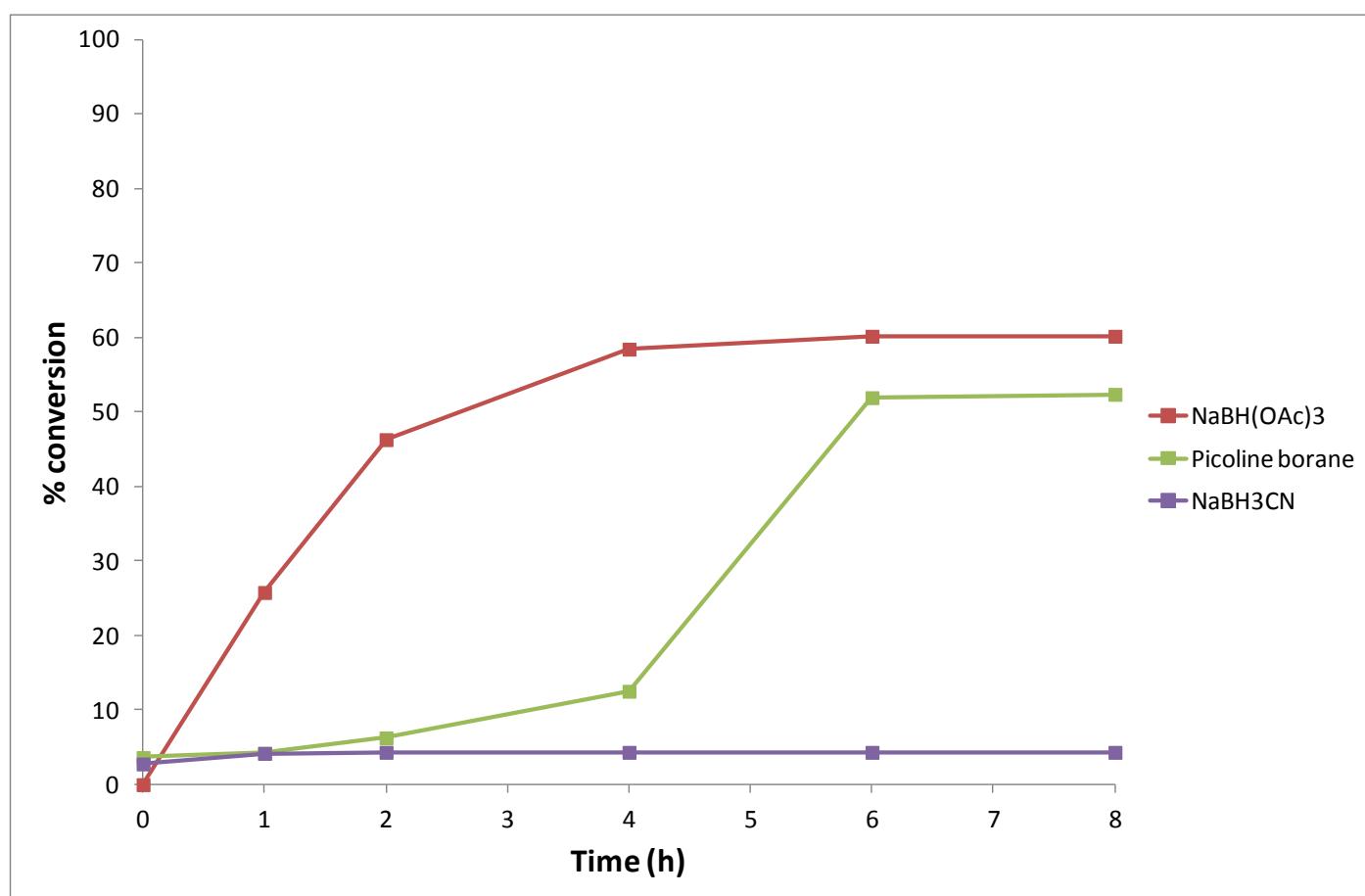
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	0.0	3.8	6.5
1	38.9	4.0	8.6
2	56.7	14.4	8.8
4	62.2	16.2	8.9
6	62.9	56.9	9.1
8	63.0	56.9	9.4
24	63.7	56.9	9.4

Reaction 9: DMF



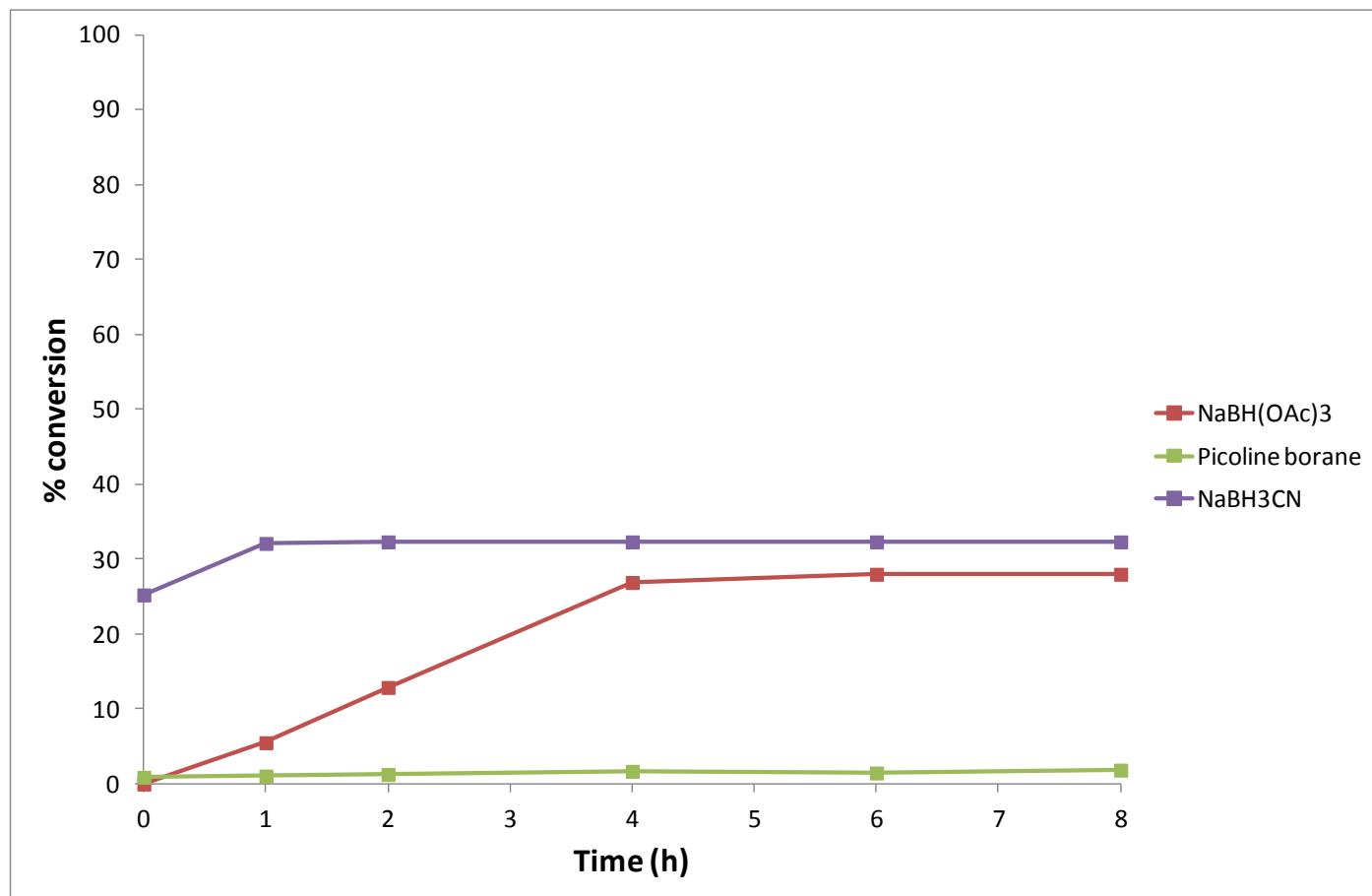
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	0.0	0.4	4.5
1	0.0	0.6	5.2
2	0.0	0.9	6.8
4	1.0	1.0	10.7
6	3.8	1.8	14.7
8	7.9	2.0	22.1
24	32.5	59.4	22.1

Reaction 9: EtOAc



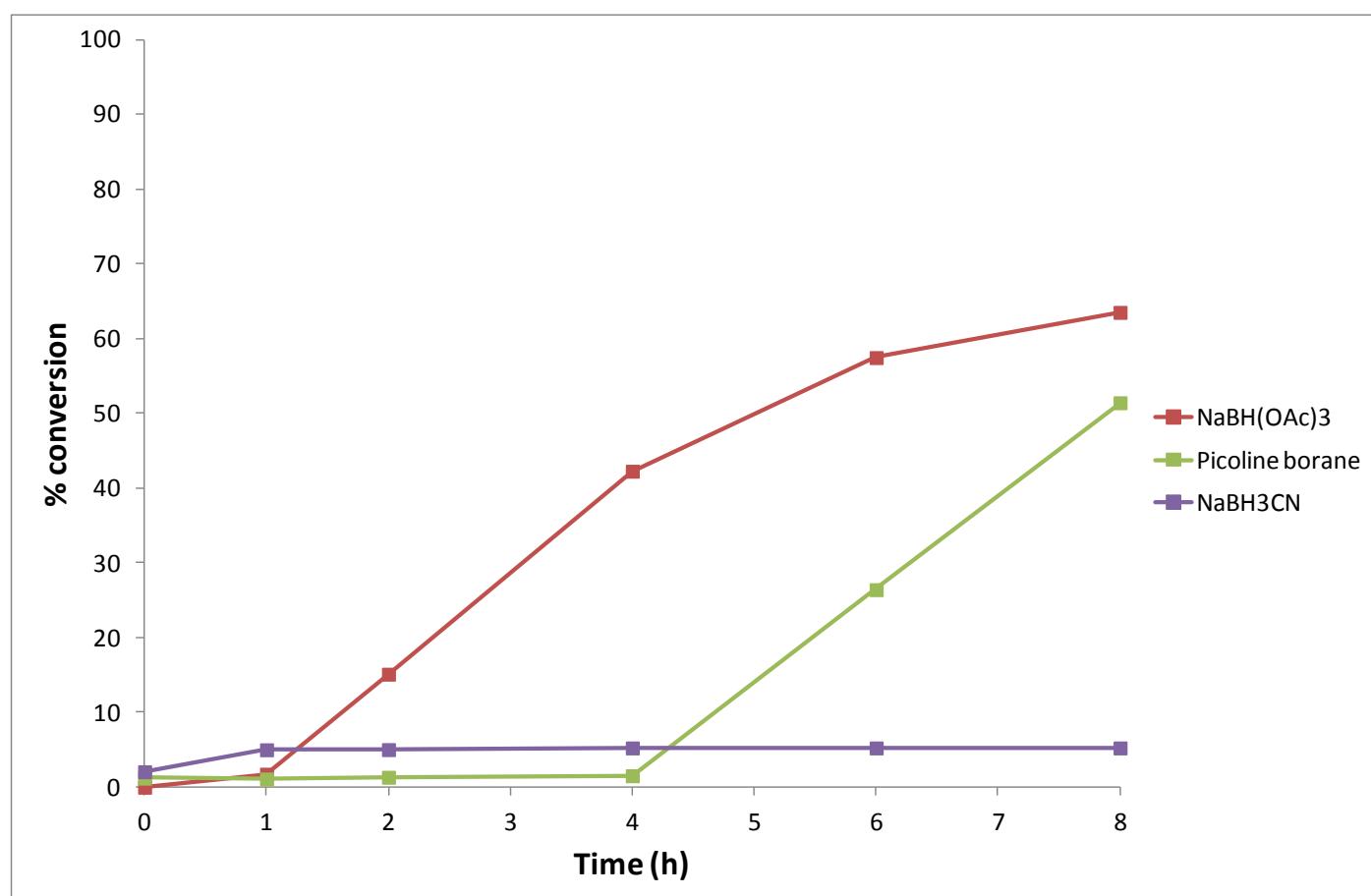
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	0.0	3.6	2.8
1	25.8	4.2	4.2
2	46.3	6.3	4.3
4	58.4	12.5	4.3
6	60.2	51.9	4.3
8	60.2	52.4	4.3
24	60.2	52.4	4.3

Reaction 9: IPA



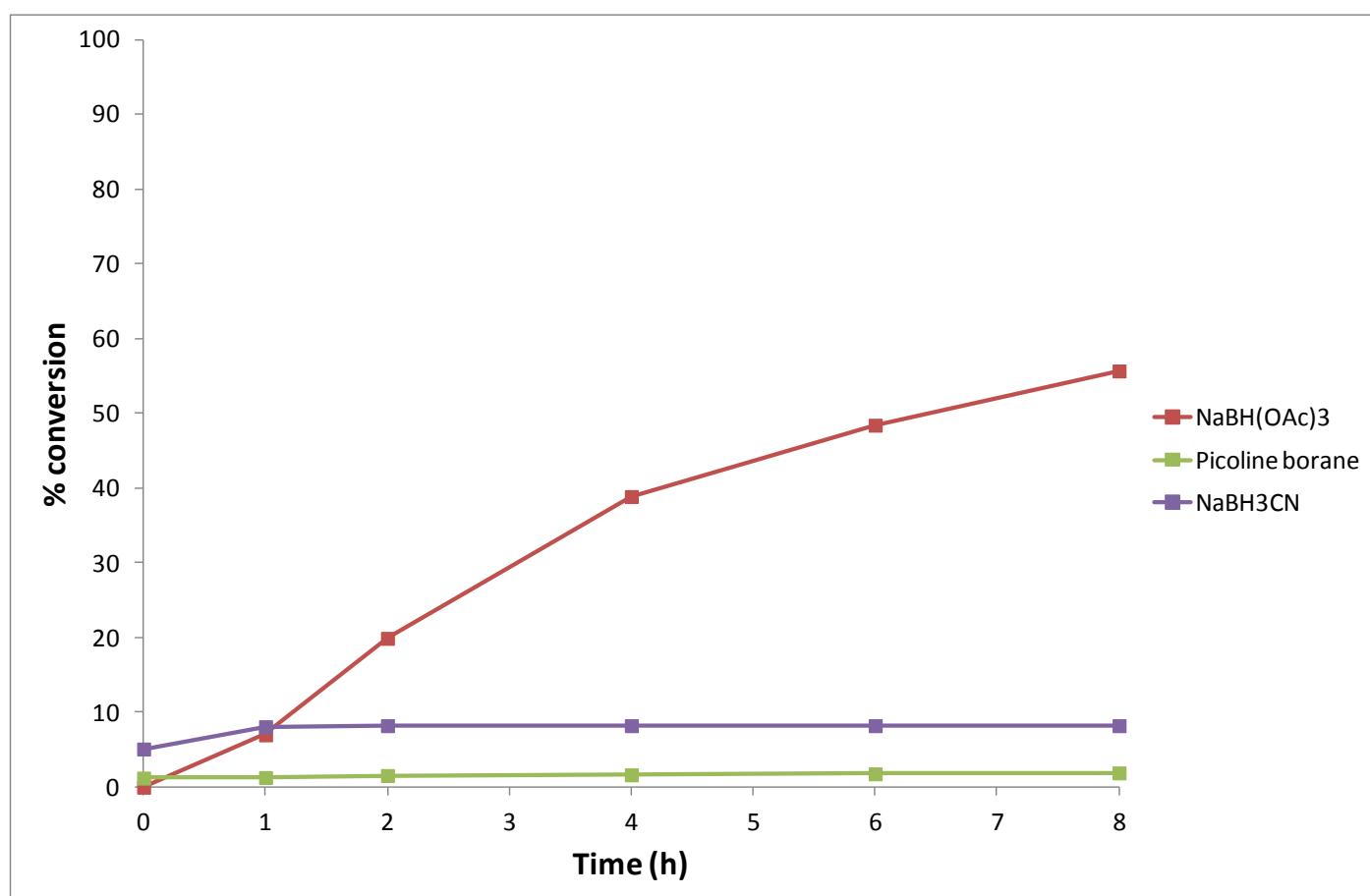
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	0.0	0.9	25.2
1	5.5	1.1	32.1
2	12.9	1.3	32.3
4	26.9	1.7	32.3
6	28.0	1.5	32.3
8	28.0	1.9	32.3
24	28.0	71.4	32.3

Reaction 9: 2-MeTHF



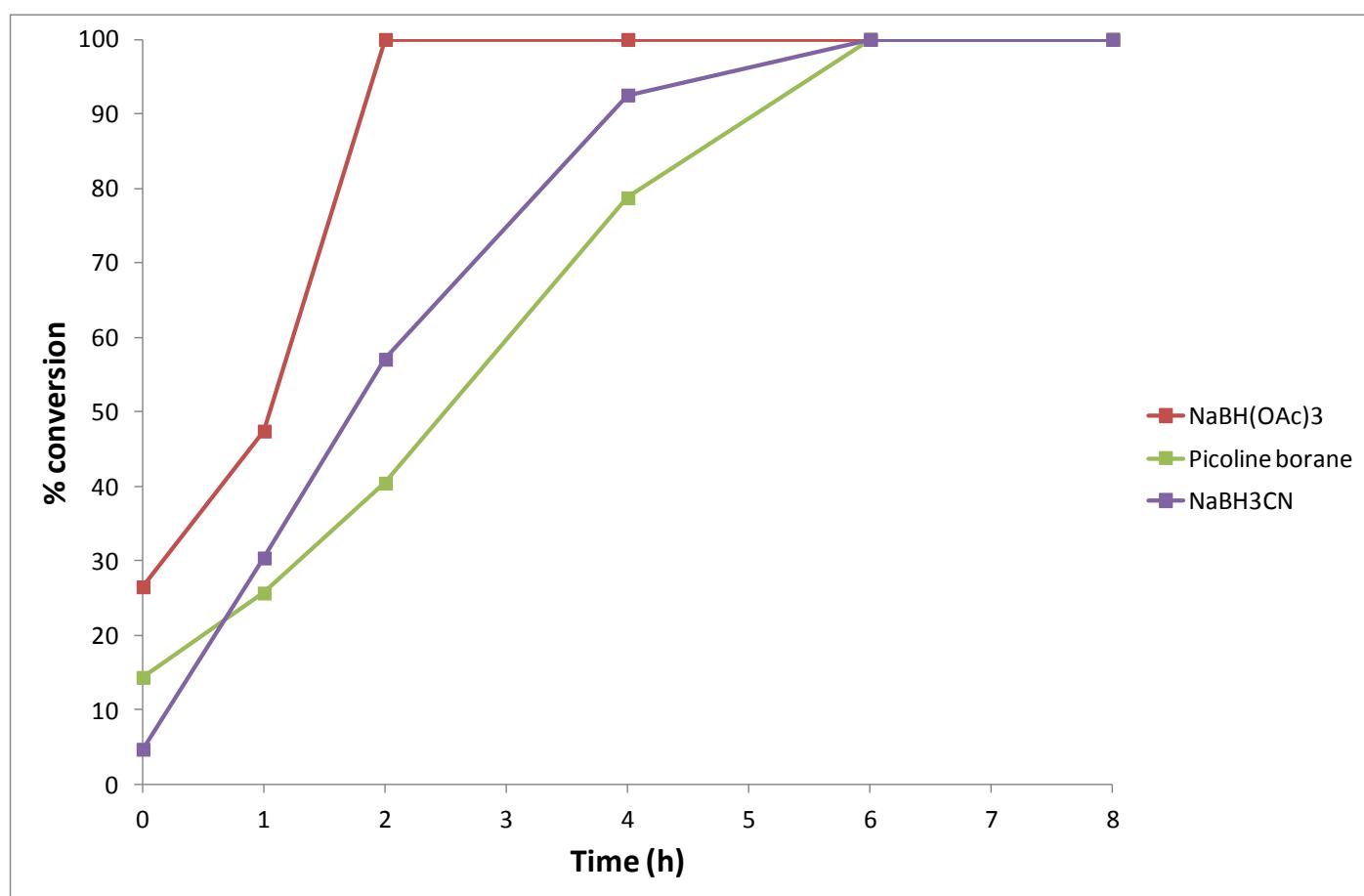
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	0.0	1.0	2.1
1	1.7	1.2	5.0
2	15.1	1.3	5.0
4	42.3	1.5	5.3
6	57.5	26.4	5.3
8	63.5	51.4	5.3
24	76.6	51.4	5.3

Reaction 9: THF



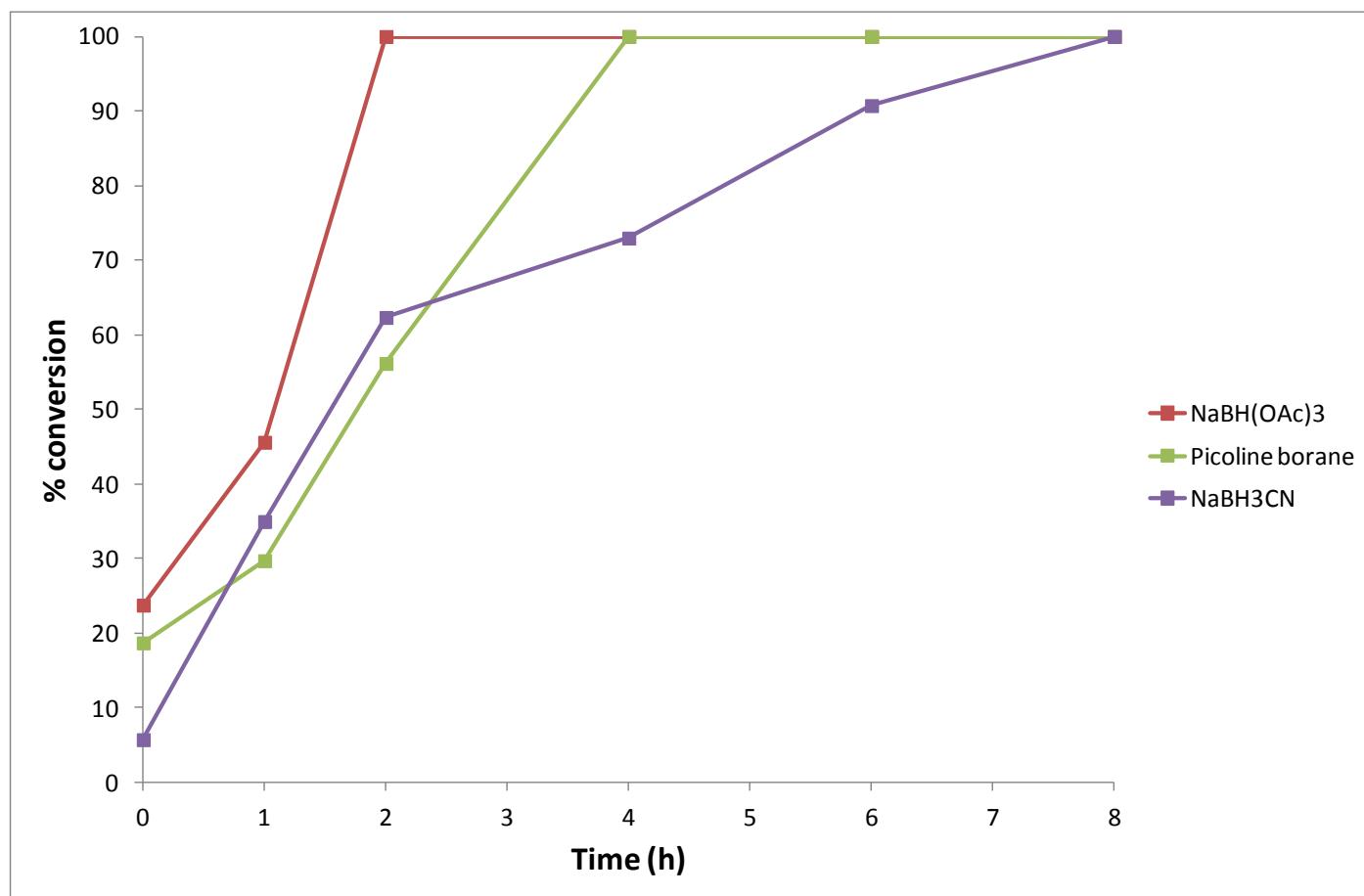
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	0.0	1.2	5.1
1	7.0	1.3	8.1
2	19.9	1.6	8.2
4	38.9	1.7	8.2
6	48.4	1.8	8.2
8	55.7	1.9	8.2
24	69.1	52.6	8.2

Reaction 10: TBME



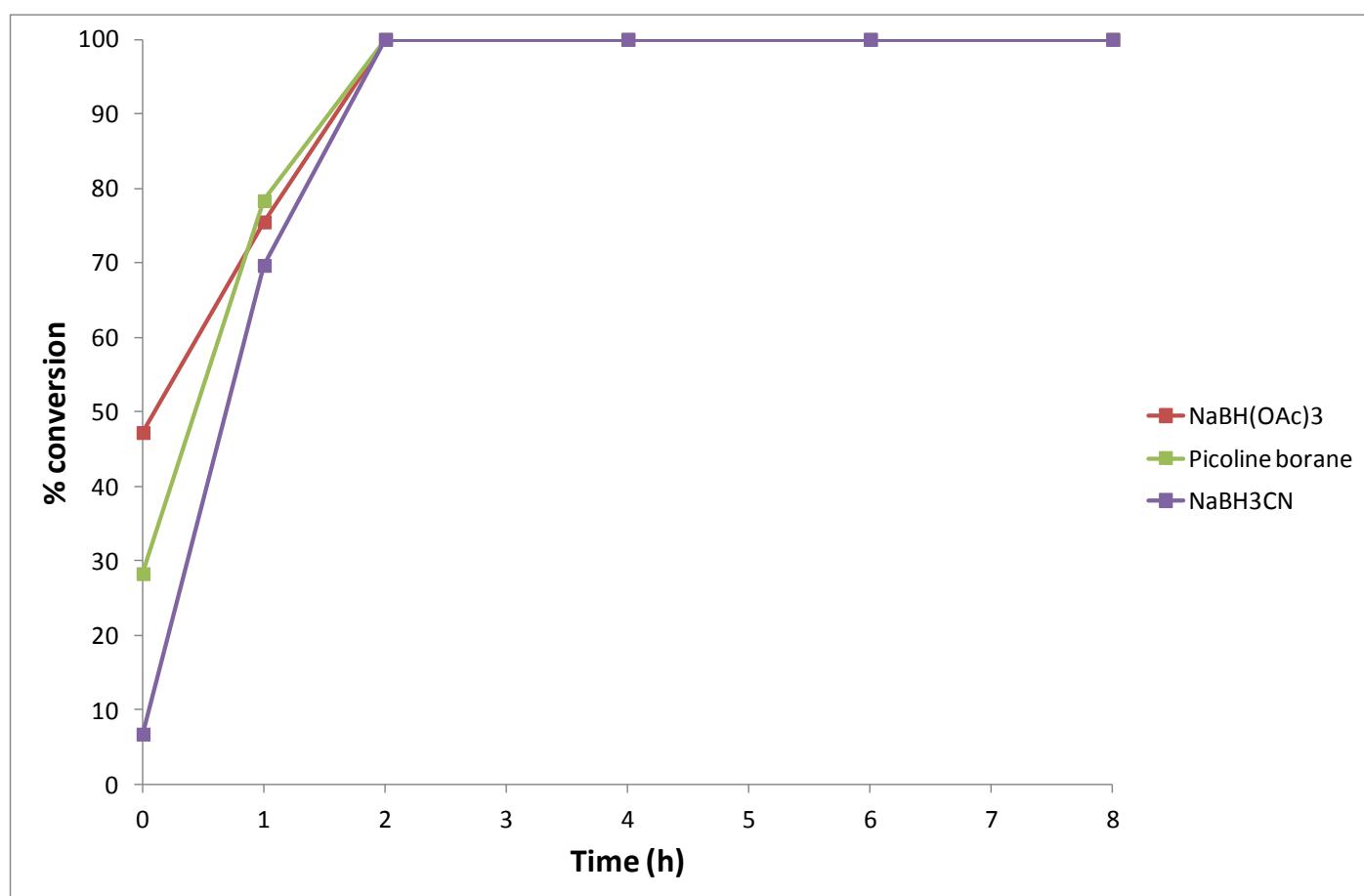
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	26.6	14.4	4.8
1	47.4	25.7	30.4
2	100.0	40.5	57.1
4	100.0	78.8	92.5
6	100.0	100.0	100.0
8	100.0	100.0	100.0
24	100.0	100.0	100.0

Reaction 10: CPME



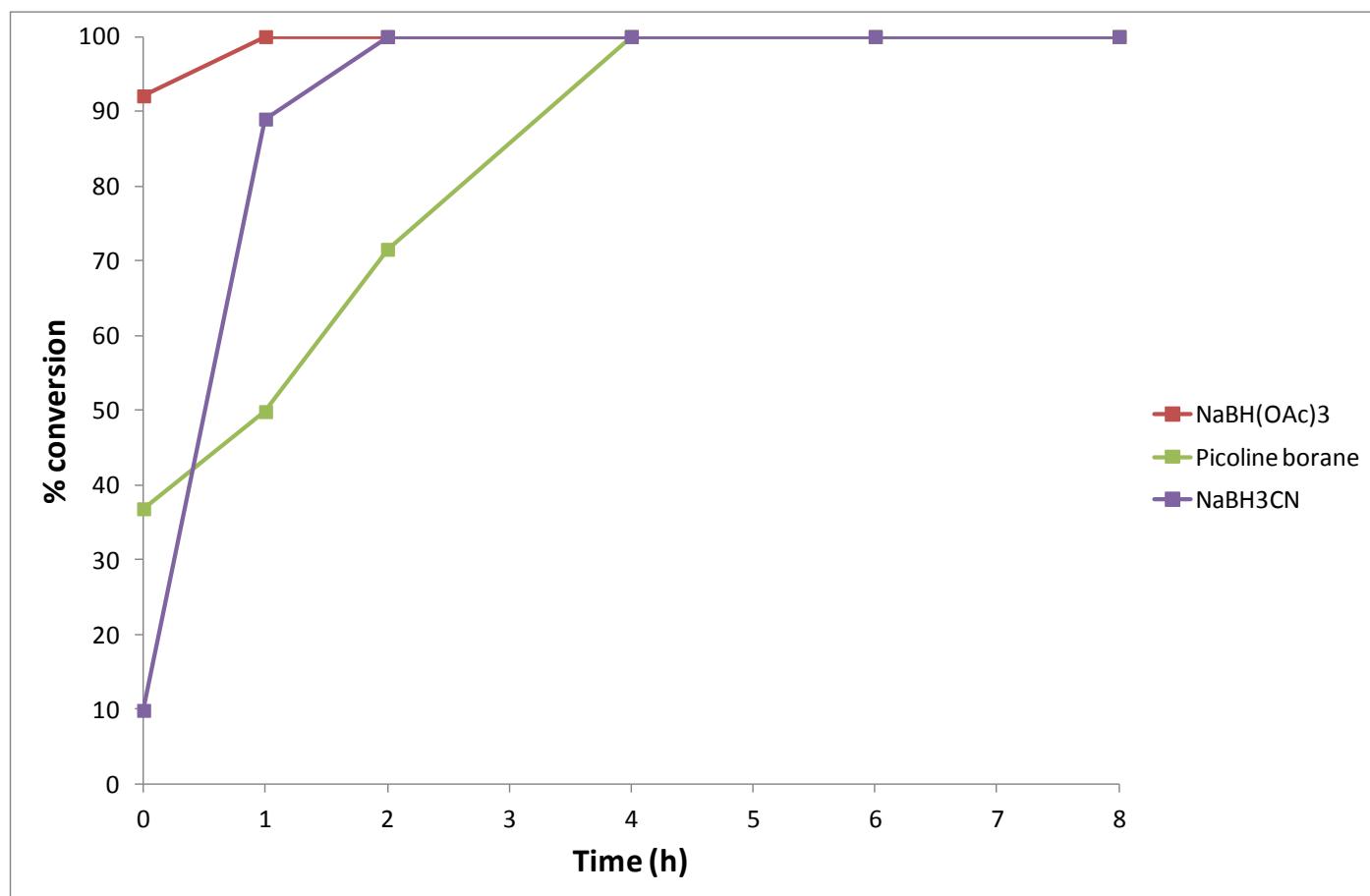
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	23.8	18.7	5.8
1	45.6	29.7	35.0
2	100.0	56.2	62.3
4	100.0	100.0	73.0
6	100.0	100.0	90.7
8	100.0	100.0	100.0
24	100.0	100.0	100.0

Reaction 10: DCE



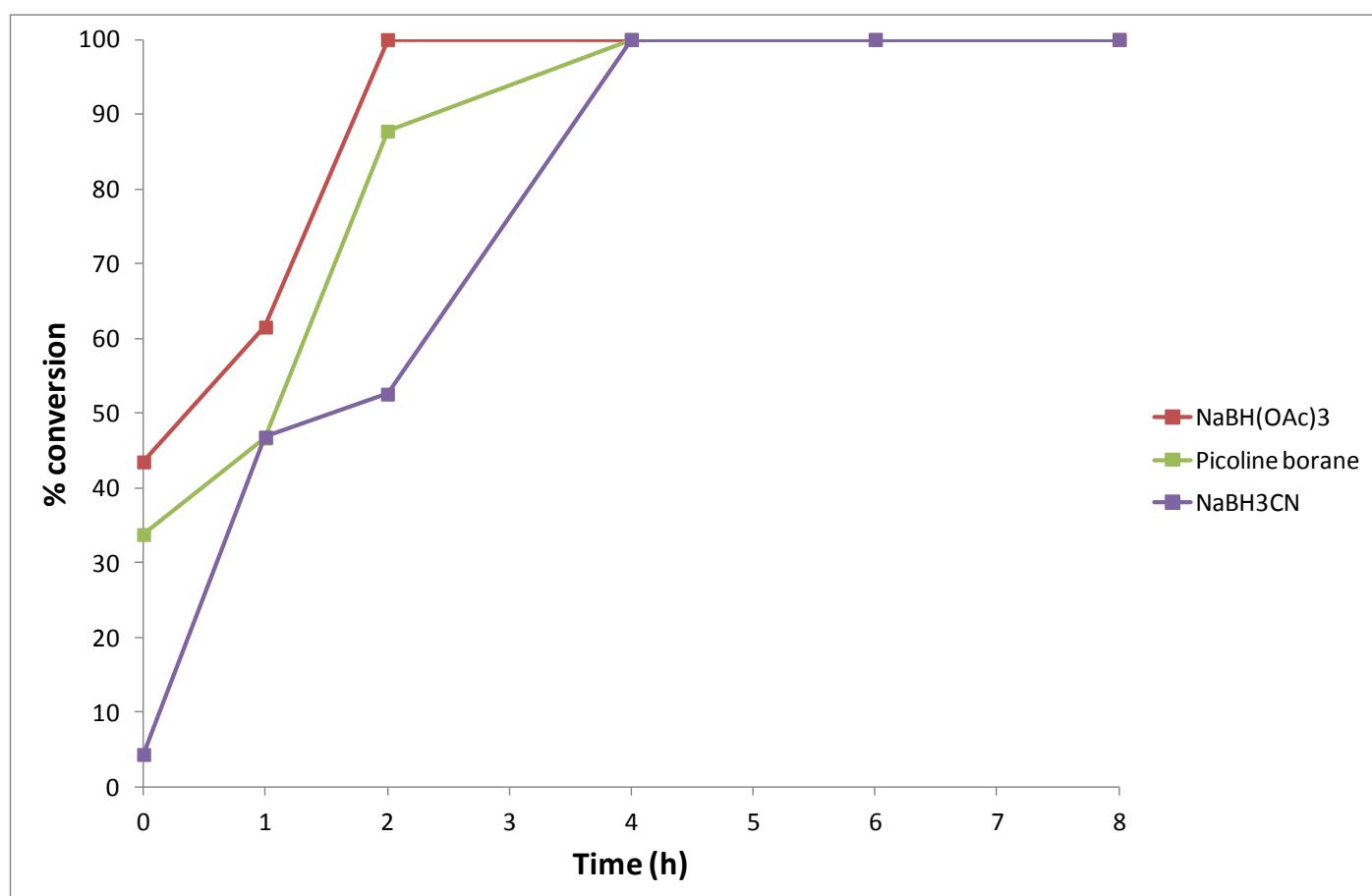
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	47.3	28.3	6.8
1	75.5	78.4	69.7
2	100.0	100.0	100.0
4	100.0	100.0	100.0
6	100.0	100.0	100.0
8	100.0	100.0	100.0
24	100.0	100.0	100.0

Reaction 10: CH₂Cl₂



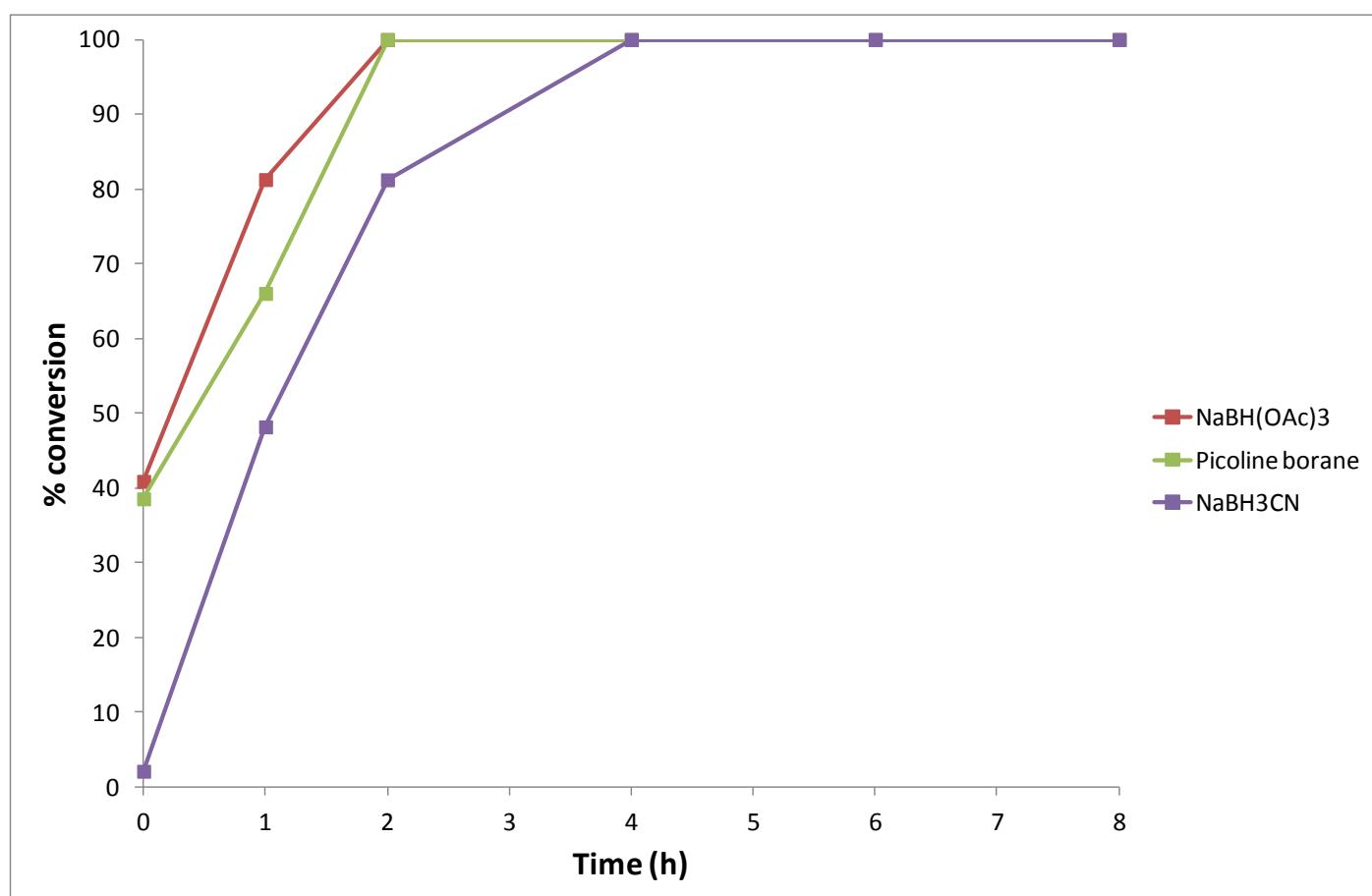
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	92.1	36.9	9.9
1	100.0	49.8	89.0
2	100.0	71.6	100.0
4	100.0	100.0	100.0
6	100.0	100.0	100.0
8	100.0	100.0	100.0
24	100.0	100.0	100.0

Reaction 10: DMC



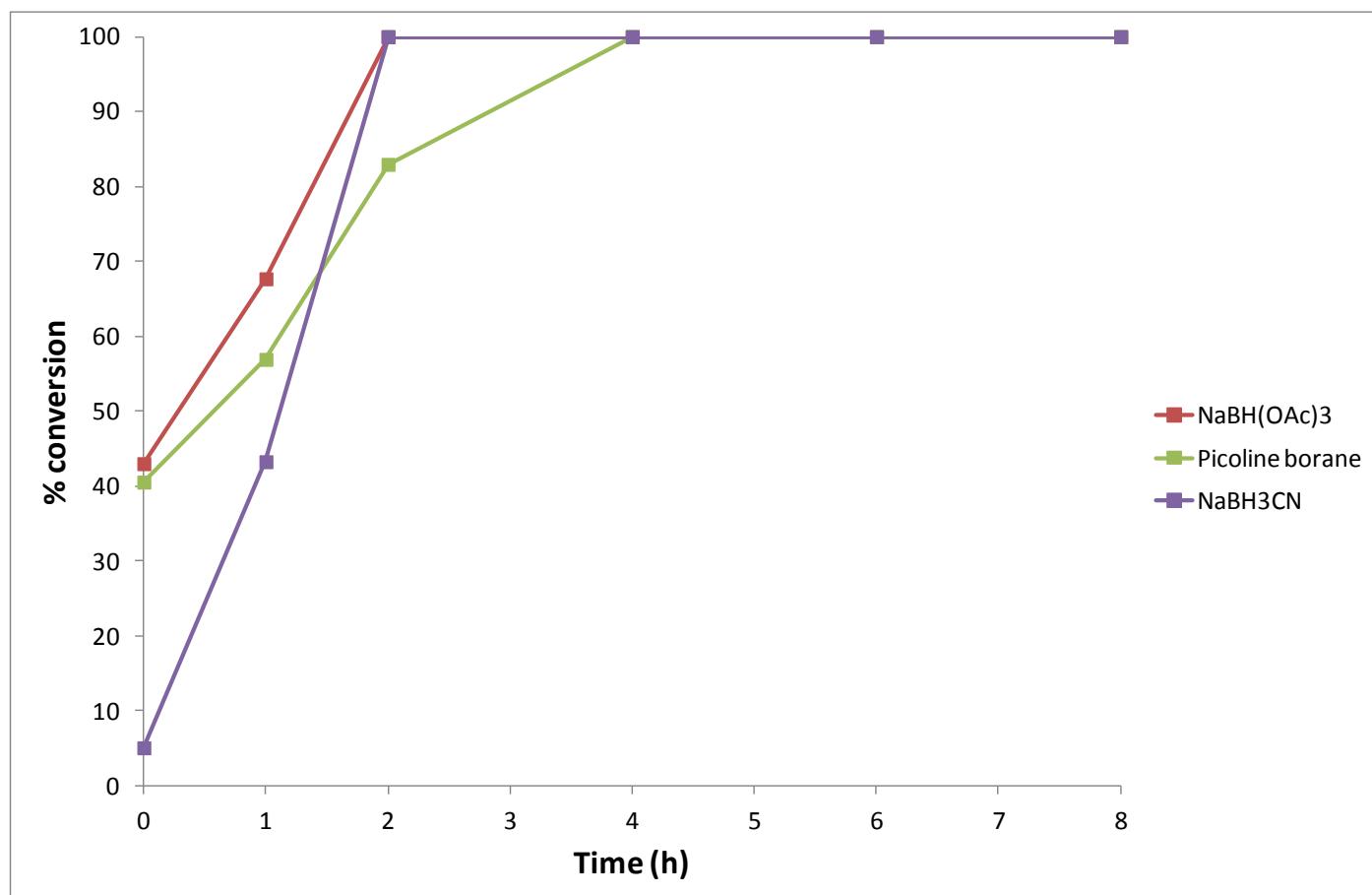
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	43.5	33.8	4.4
1	61.5	46.7	46.8
2	100.0	87.8	52.6
4	100.0	100.0	100.0
6	100.0	100.0	100.0
8	100.0	100.0	100.0
24	100.0	100.0	100.0

Reaction 10: DMF



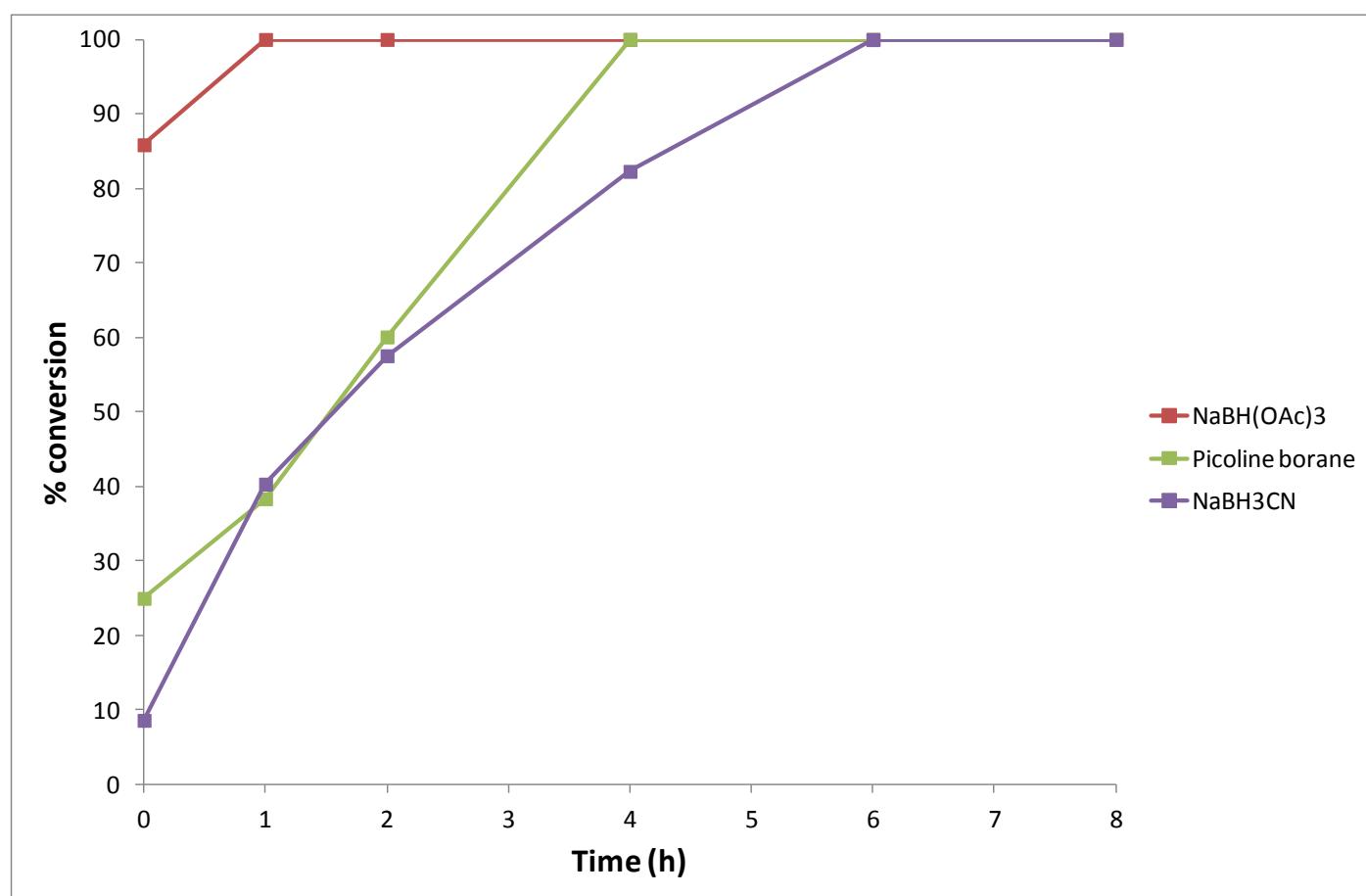
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	40.9	38.6	2.1
1	81.3	66.0	48.2
2	100.0	100.0	81.2
4	100.0	100.0	100.0
6	100.0	100.0	100.0
8	100.0	100.0	100.0
24	100.0	100.0	100.0

Reaction 10: EtOAc



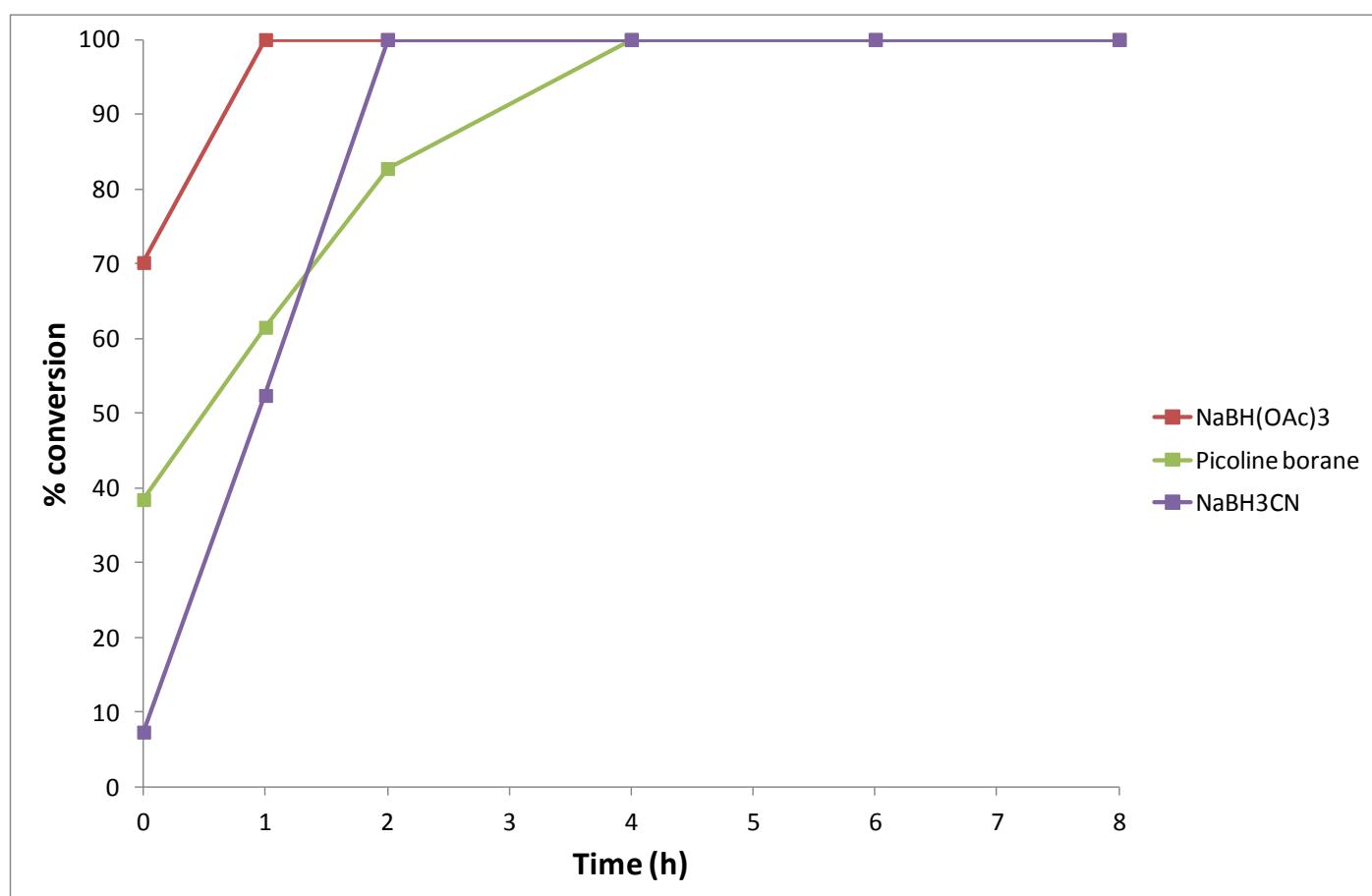
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	43.0	40.6	5.1
1	67.7	56.9	43.3
2	100.0	83.0	100.0
4	100.0	100.0	100.0
6	100.0	100.0	100.0
8	100.0	100.0	100.0
24	100.0	100.0	100.0

Reaction 10: IPA



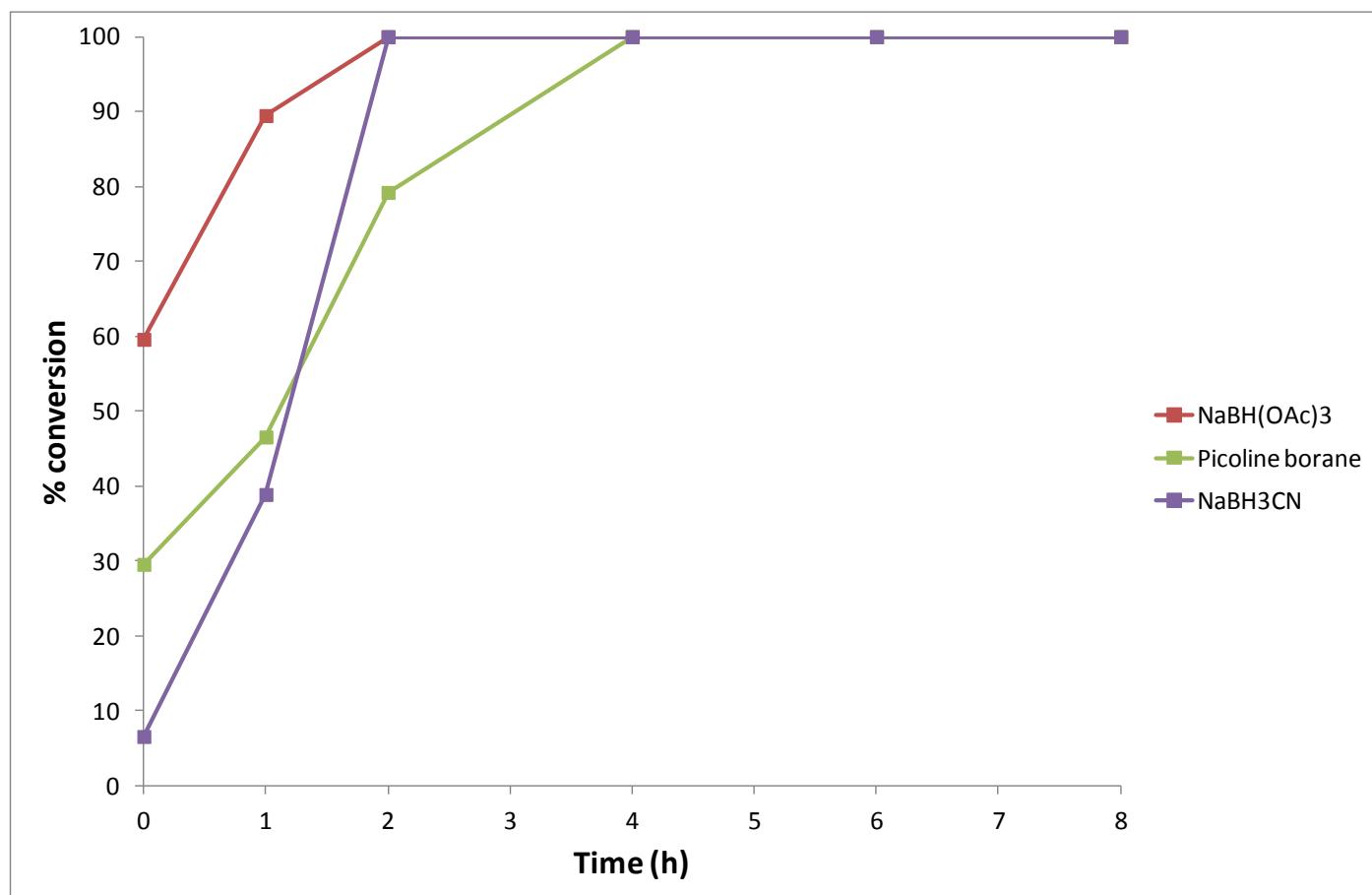
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	85.9	25.0	8.6
1	100.0	38.4	40.3
2	100.0	60.1	57.6
4	100.0	100.0	82.3
6	100.0	100.0	100.0
8	100.0	100.0	100.0
24	100.0	100.0	100.0

Reaction 10: 2-MeTHF



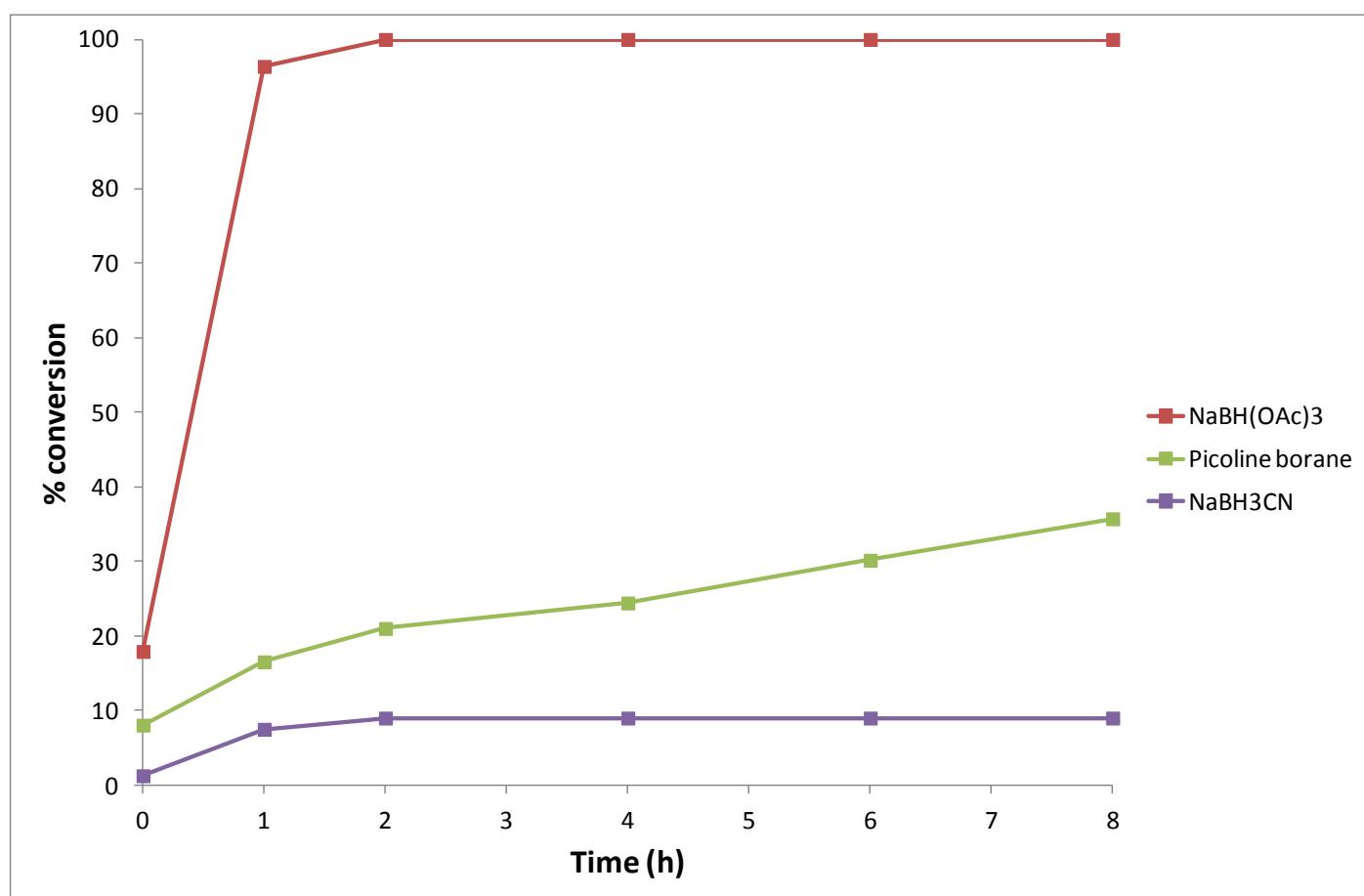
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	70.2	38.5	7.4
1	100.0	61.5	52.4
2	100.0	82.8	100.0
4	100.0	100.0	100.0
6	100.0	100.0	100.0
8	100.0	100.0	100.0
24	100.0	100.0	100.0

Reaction 10: THF



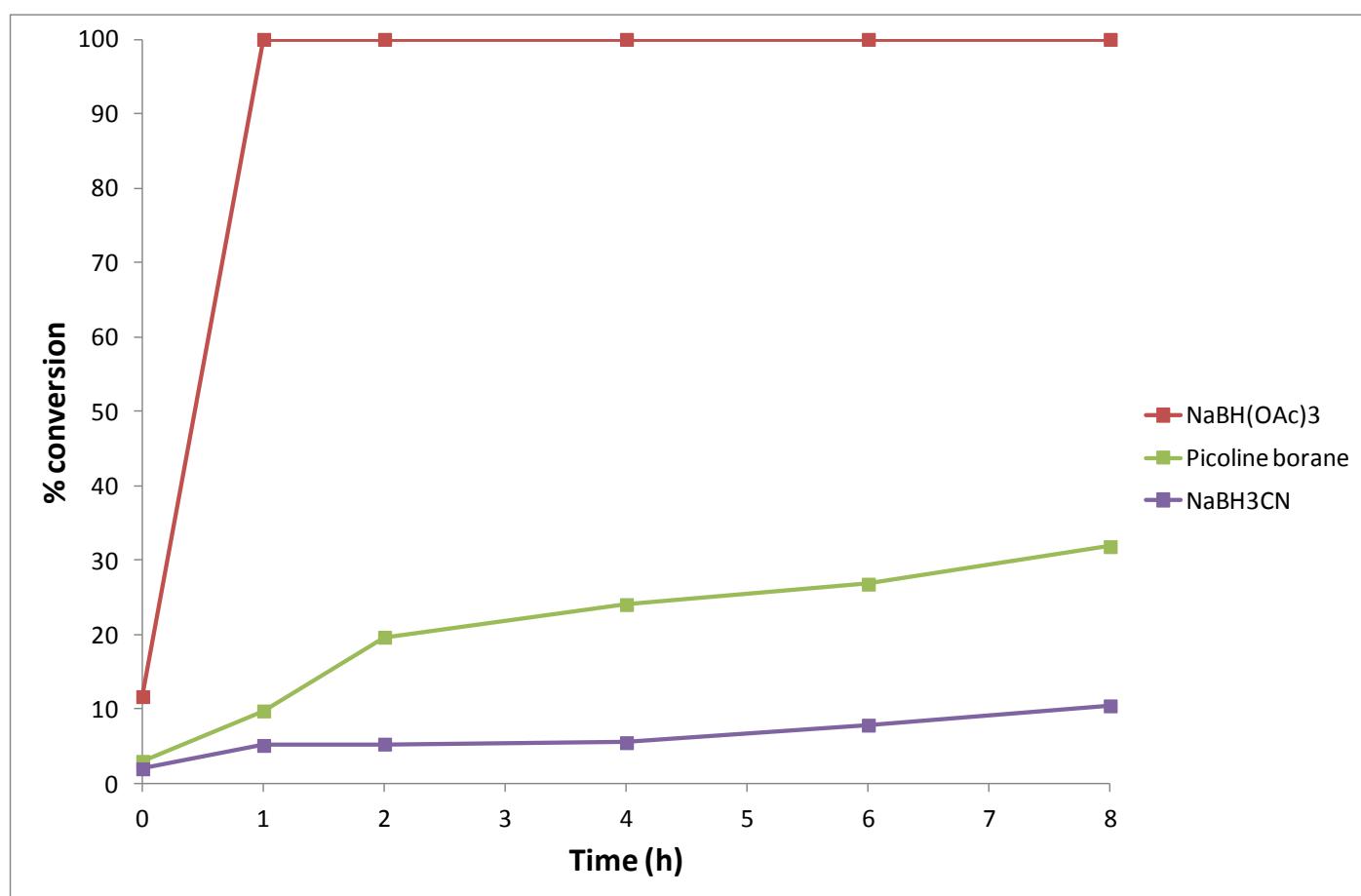
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	59.6	29.6	6.6
1	89.5	46.6	38.9
2	100.0	79.2	100.0
4	100.0	100.0	100.0
6	100.0	100.0	100.0
8	100.0	100.0	100.0
24	100.0	100.0	100.0

Reaction 11: TBME



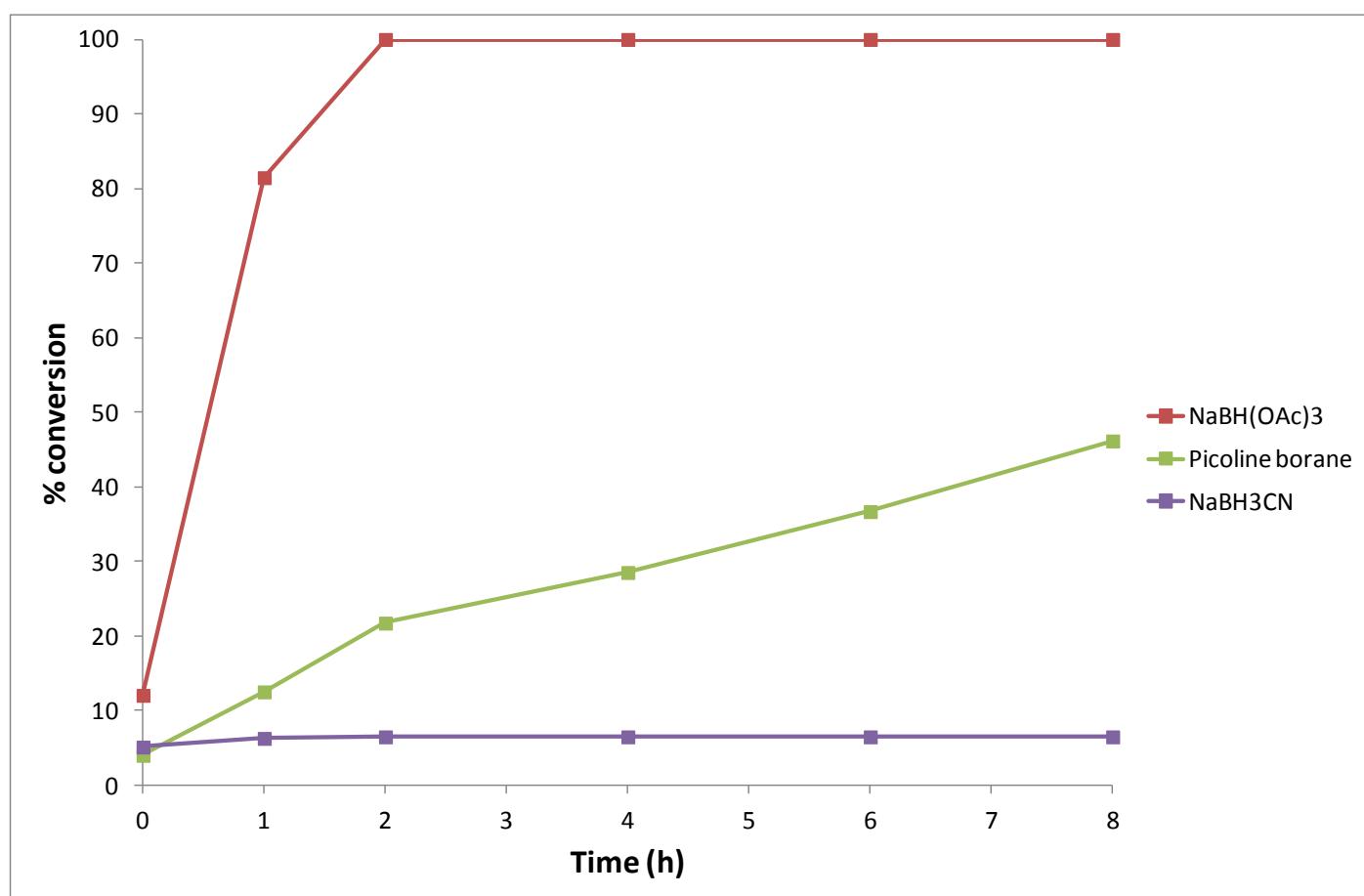
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	18.0	8.1	0.0
1	96.4	16.6	1.3
2	100.0	21.0	7.5
4	100.0	24.5	9.0
6	100.0	30.2	9.0
8	100.0	35.8	9.0
24	100.0	40.8	9.0

Reaction 11: CPME



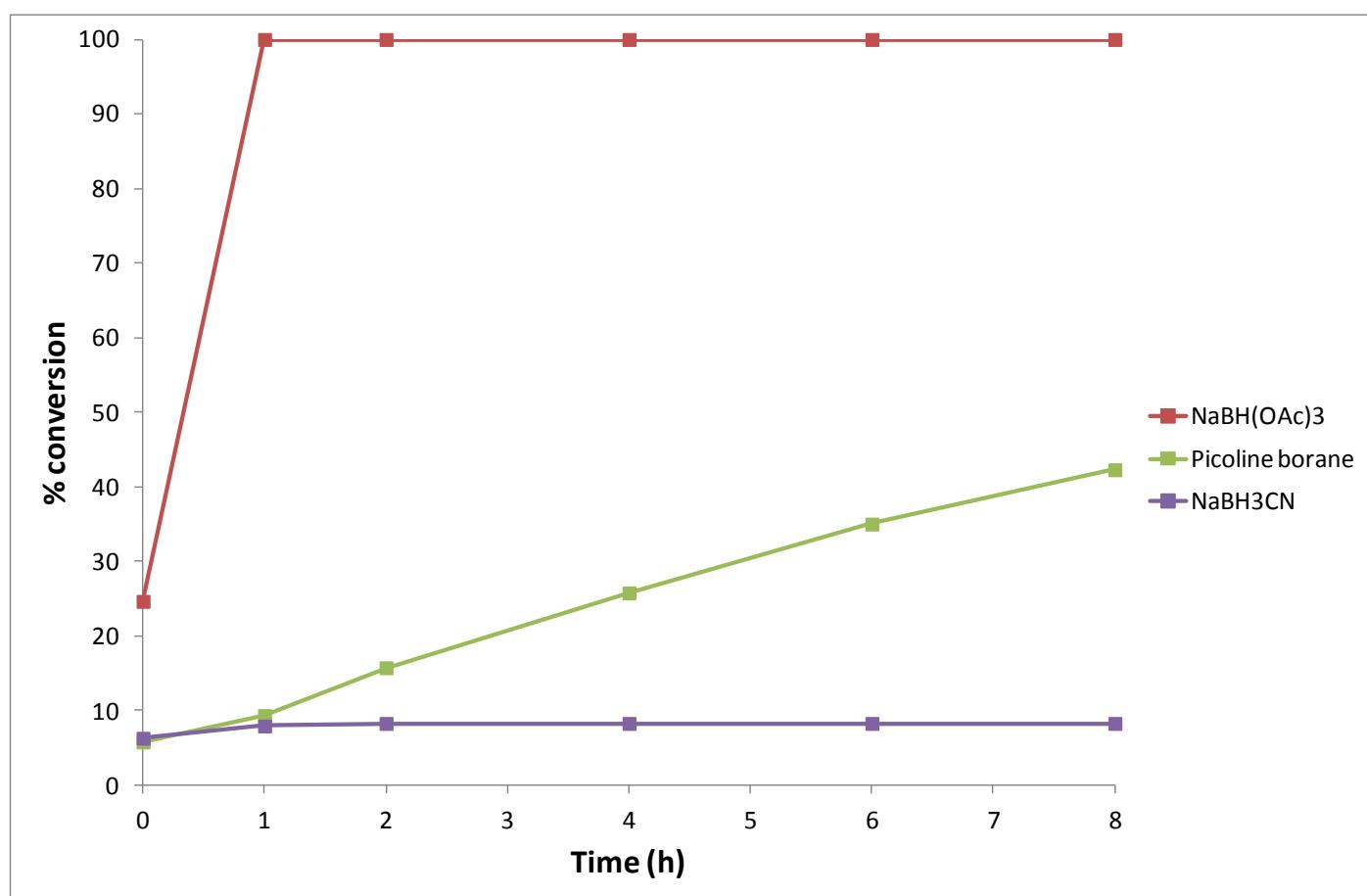
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	11.7	3.0	0.0
1	100.0	9.8	2.0
2	100.0	19.7	5.2
4	100.0	24.1	5.3
6	100.0	26.8	5.5
8	100.0	31.9	7.9
24	100.0	38.4	10.5

Reaction 11: DCE



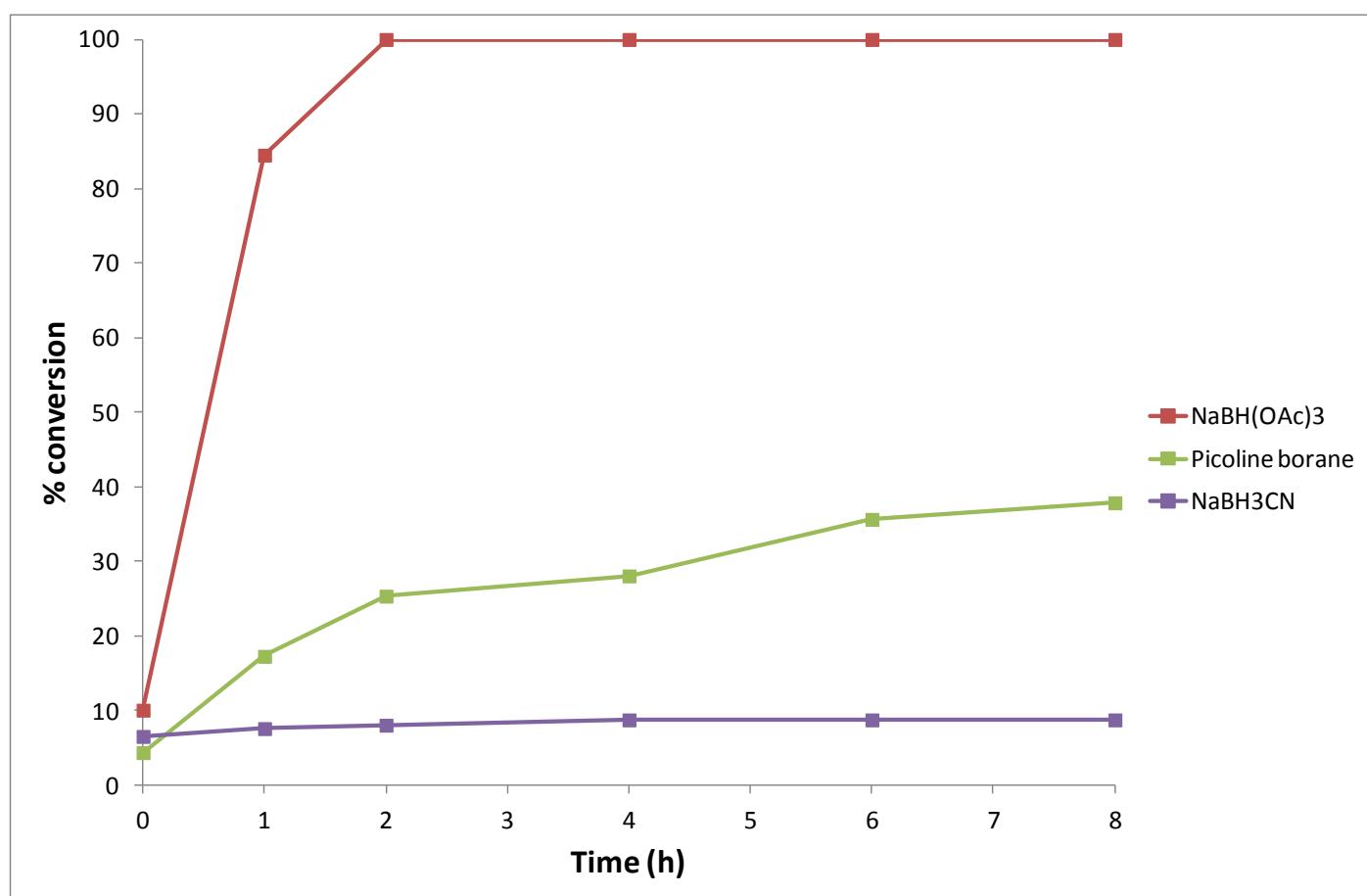
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	12.1	4.0	0.0
1	81.5	12.5	5.2
2	100.0	21.8	6.3
4	100.0	28.5	6.5
6	100.0	36.7	6.5
8	100.0	46.2	6.5
24	100.0	61.1	6.5

Reaction 11: CH₂Cl₂



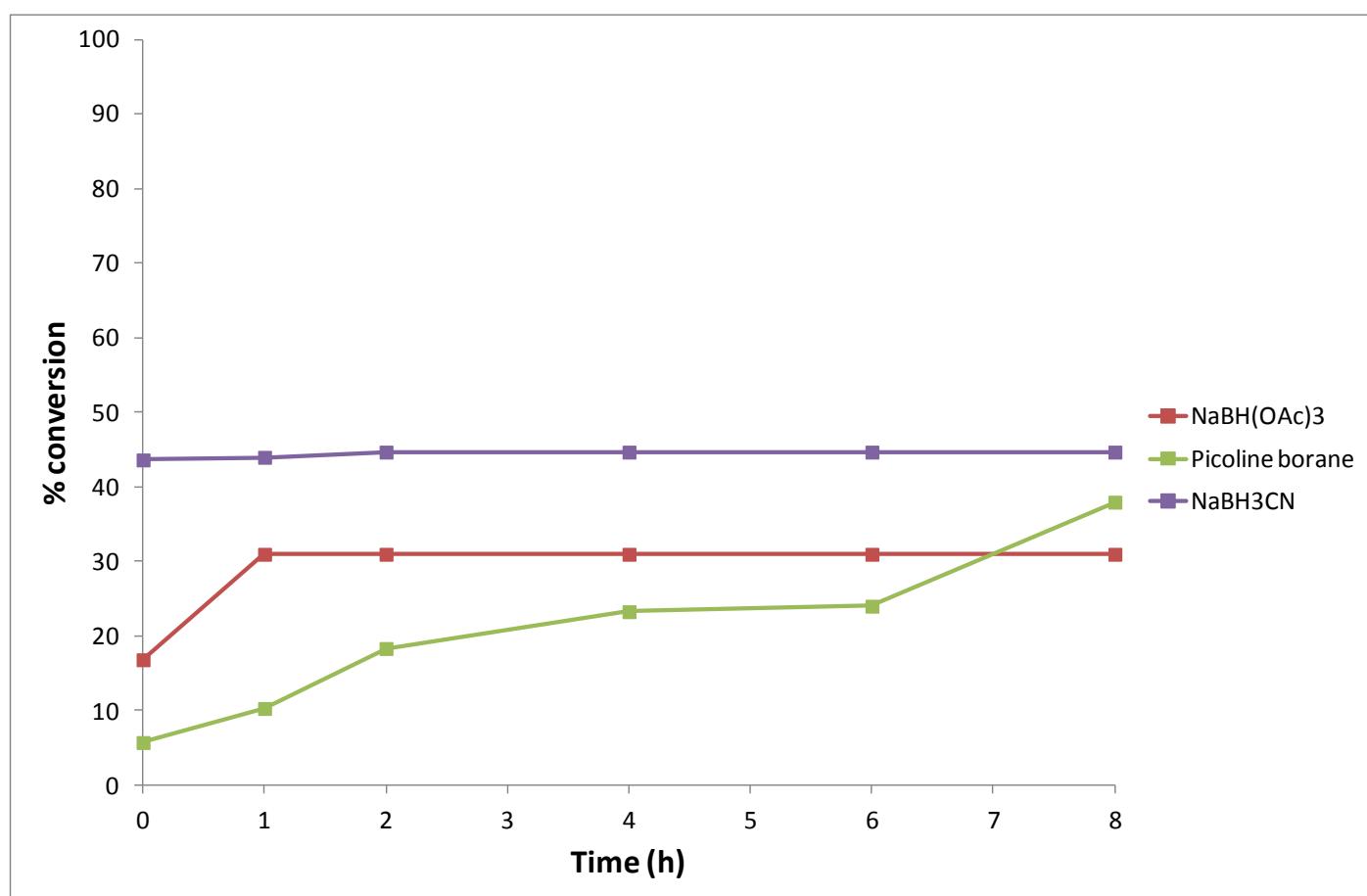
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	24.7	5.8	0.0
1	100.0	9.4	6.3
2	100.0	15.8	8.0
4	100.0	25.8	8.3
6	100.0	35.0	8.3
8	100.0	42.3	8.3
24	100.0	63.5	8.3

Reaction 11: DMC



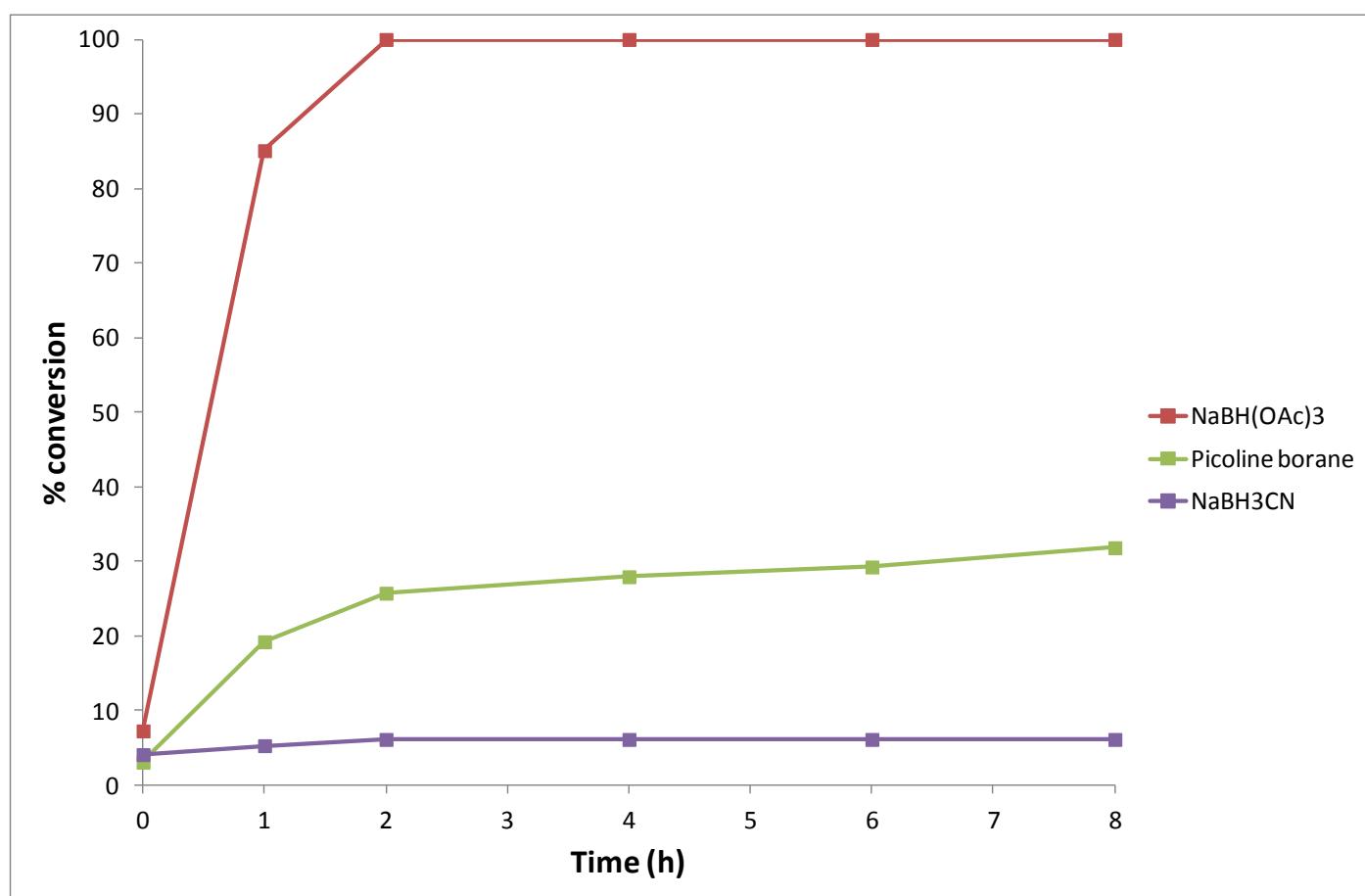
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	10.1	4.4	0.0
1	84.5	17.3	6.6
2	100.0	25.4	7.6
4	100.0	28.1	8.1
6	100.0	35.7	8.8
8	100.0	37.9	8.8
24	100.0	44.7	8.8

Reaction 11: DMF



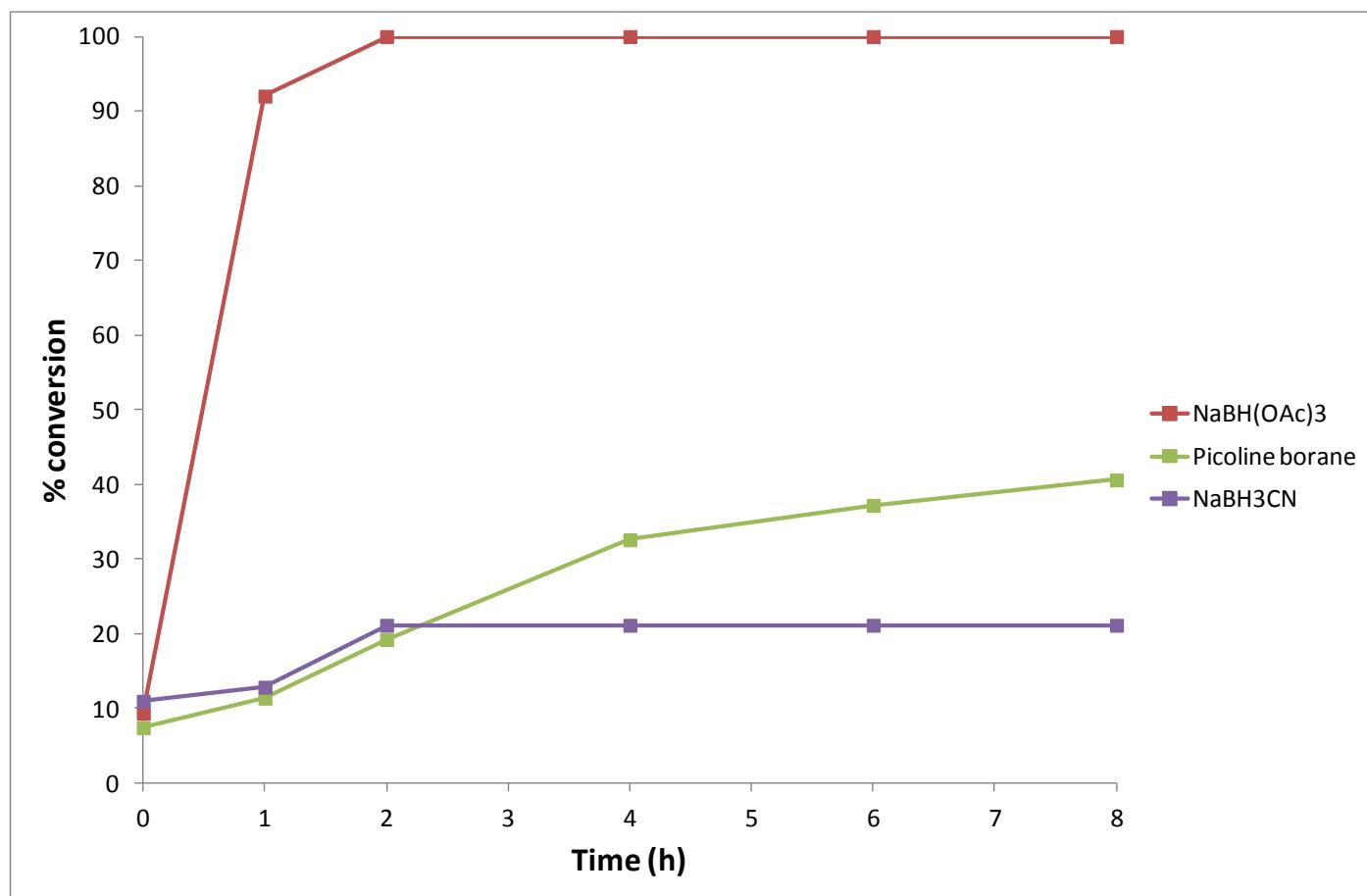
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	16.8	5.7	0.0
1	31.0	10.3	43.6
2	31.0	18.3	44.0
4	31.0	23.3	44.7
6	31.0	24.0	44.7
8	31.0	38.0	44.7
24	31.0	63.9	44.7

Reaction 11: EtOAc



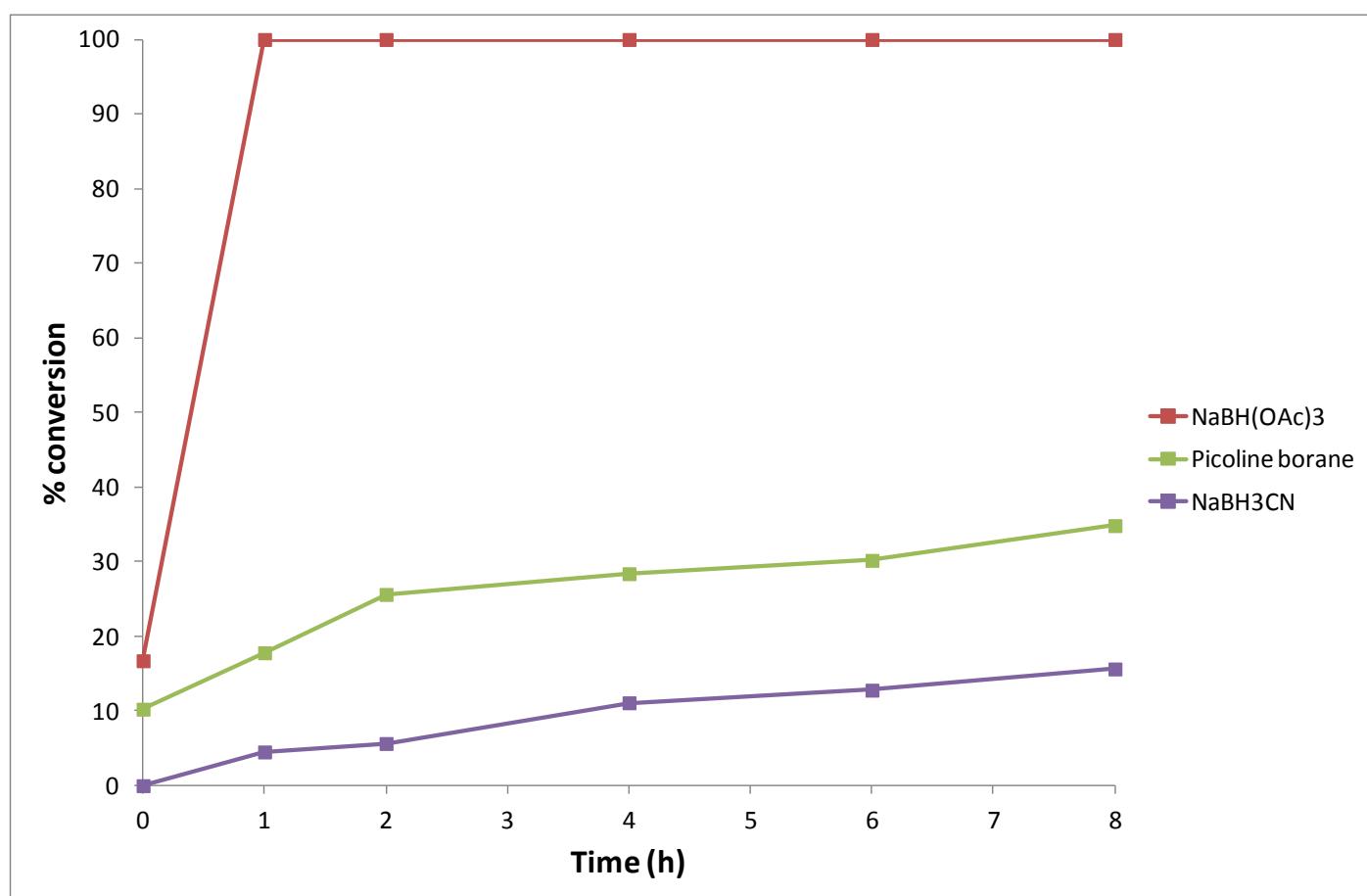
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	7.3	3.1	0.0
1	85.1	19.2	4.1
2	100.0	25.8	5.3
4	100.0	28.0	6.2
6	100.0	29.3	6.2
8	100.0	31.9	6.2
24	100.0	45.3	6.2

Reaction 11: IPA



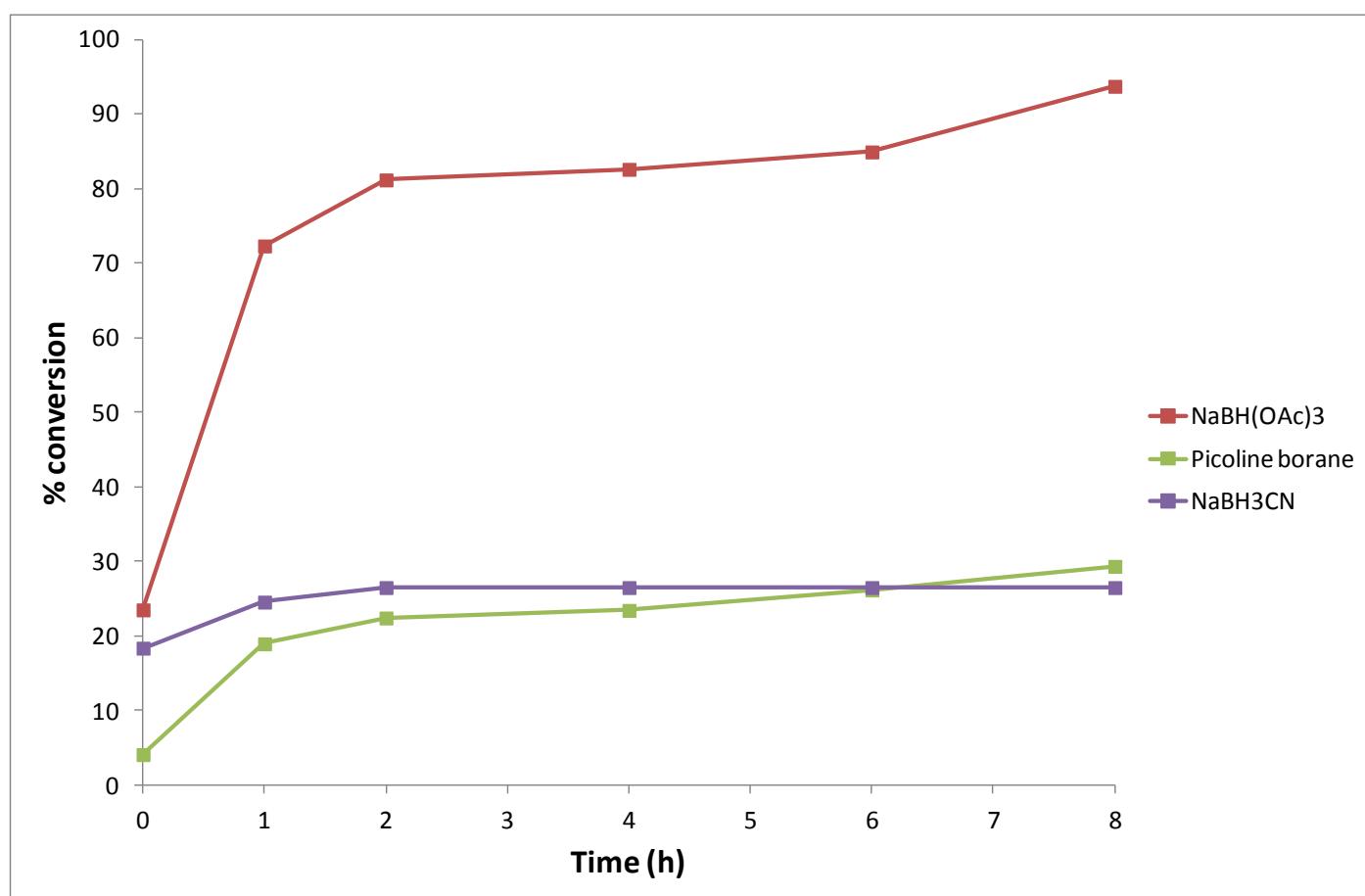
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	9.4	7.4	0.0
1	92.0	11.4	11.0
2	100.0	19.2	12.9
4	100.0	32.6	21.2
6	100.0	37.2	21.2
8	100.0	40.6	21.2
24	100.0	40.6	21.2

Reaction 11: 2-MeTHF



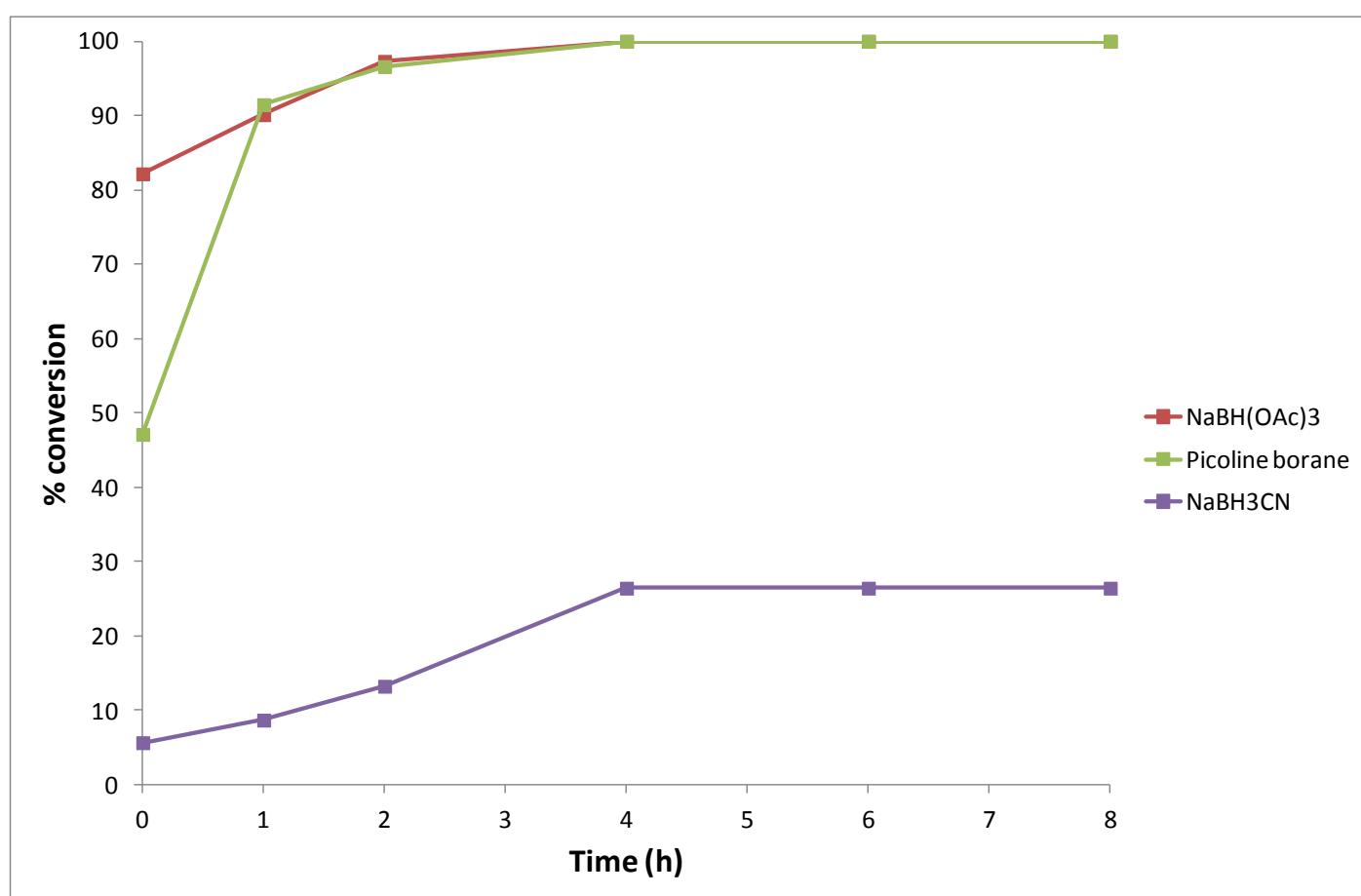
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	16.7	10.3	0.0
1	100.0	17.8	4.5
2	100.0	25.6	5.6
4	100.0	28.4	11.1
6	100.0	30.2	12.8
8	100.0	34.9	15.6
24	100.0	43.2	40.5

Reaction 11: THF



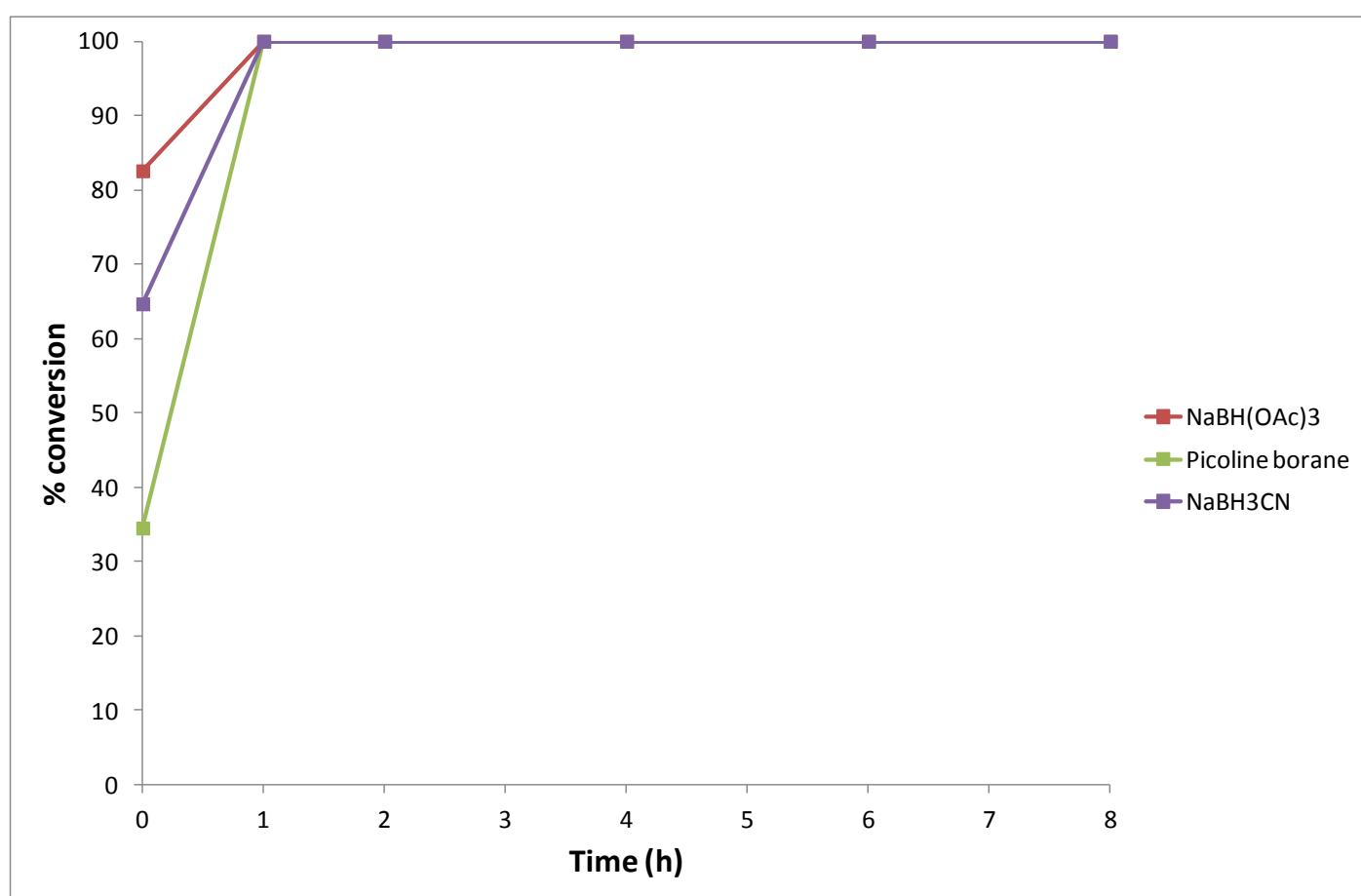
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	23.5	4.1	0.0
1	72.3	19.0	18.4
2	81.2	22.4	24.6
4	82.6	23.5	26.5
6	84.9	26.2	26.5
8	93.7	29.4	26.5
24	100.0	36.9	26.5

Reaction 12: TBME



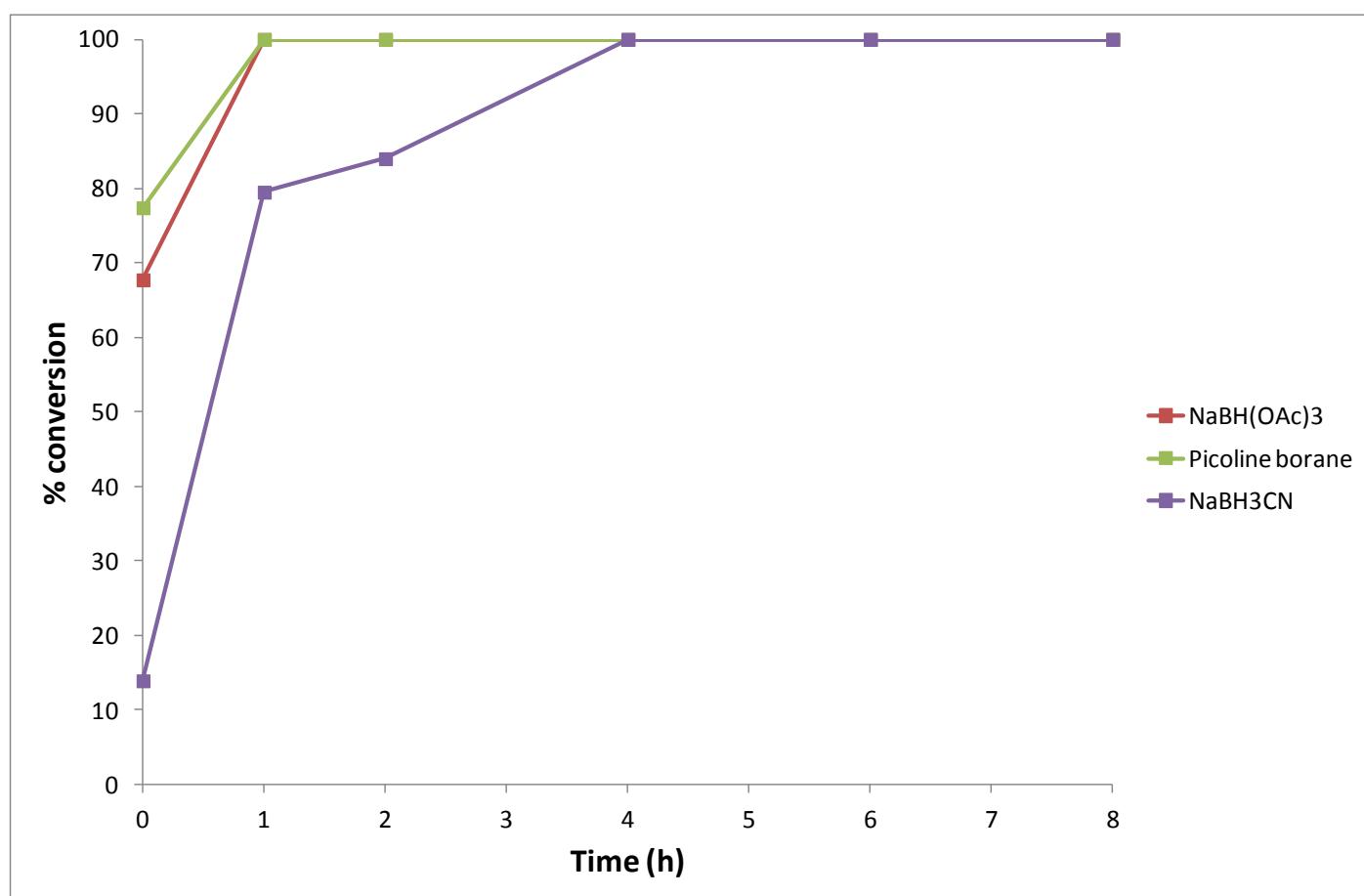
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	82.2	47.1	5.7
1	90.1	91.5	8.7
2	97.3	96.6	13.3
4	100.0	100.0	26.5
6	100.0	100.0	26.5
8	100.0	100.0	26.5
24	100.0	100.0	26.5

Reaction 12: CPME



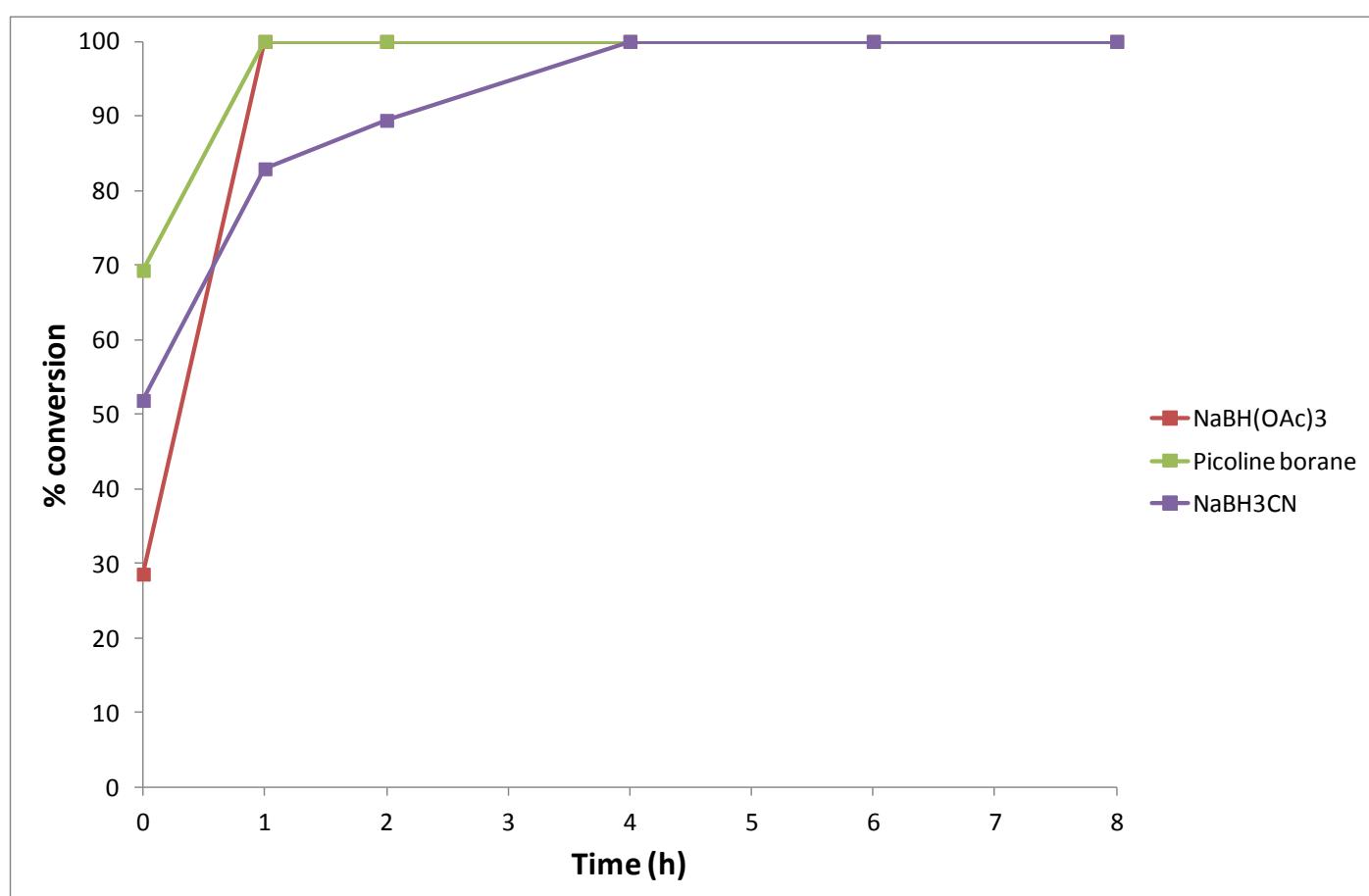
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	82.6	34.5	64.7
1	100.0	100.0	100.0
2	100.0	100.0	100.0
4	100.0	100.0	100.0
6	100.0	100.0	100.0
8	100.0	100.0	100.0
24	100.0	100.0	100.0

Reaction 12: DCE



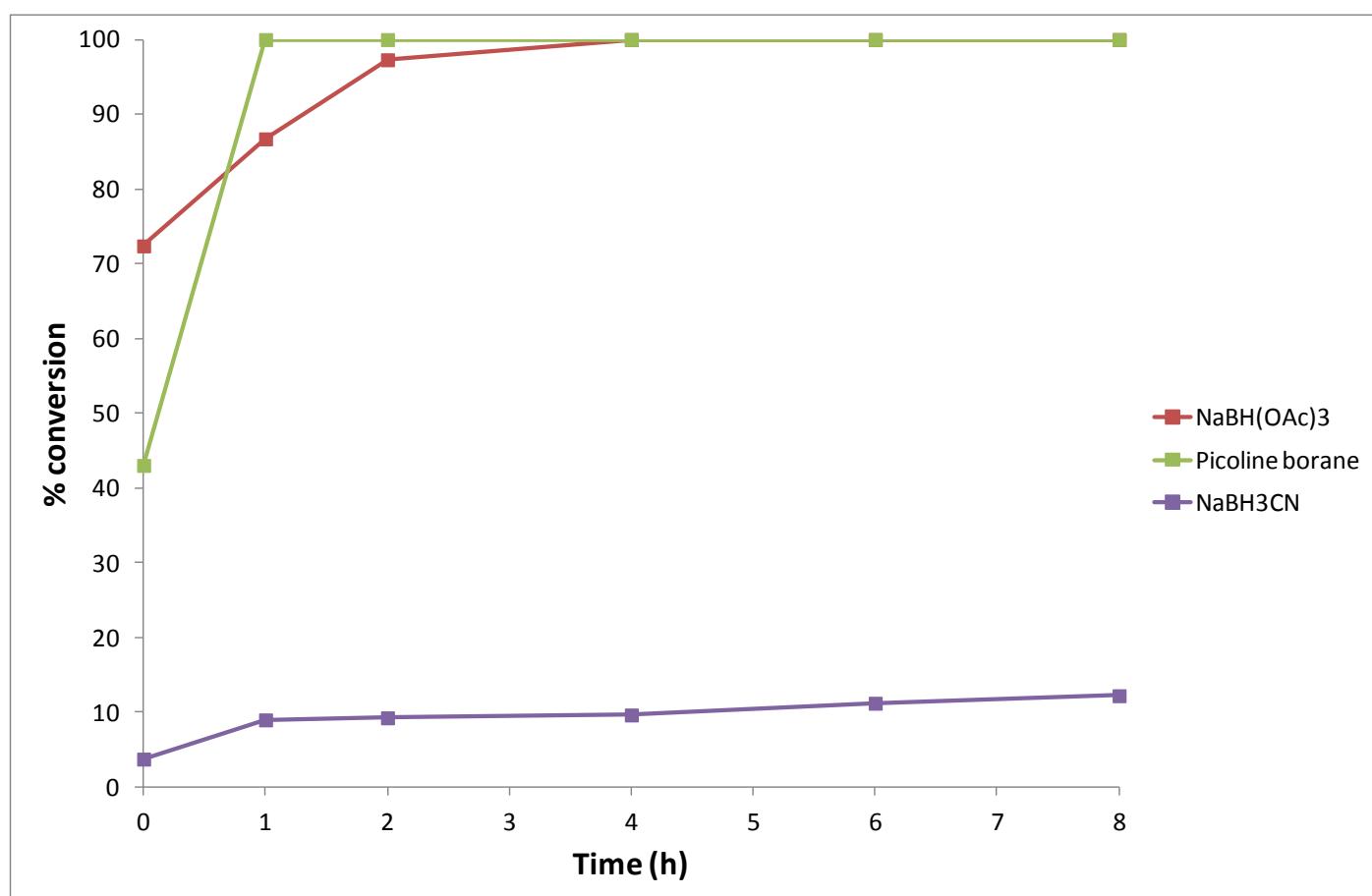
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	67.7	77.4	13.9
1	100.0	100.0	79.5
2	100.0	100.0	84.0
4	100.0	100.0	100.0
6	100.0	100.0	100.0
8	100.0	100.0	100.0
24	100.0	100.0	100.0

Reaction 12: CH₂Cl₂



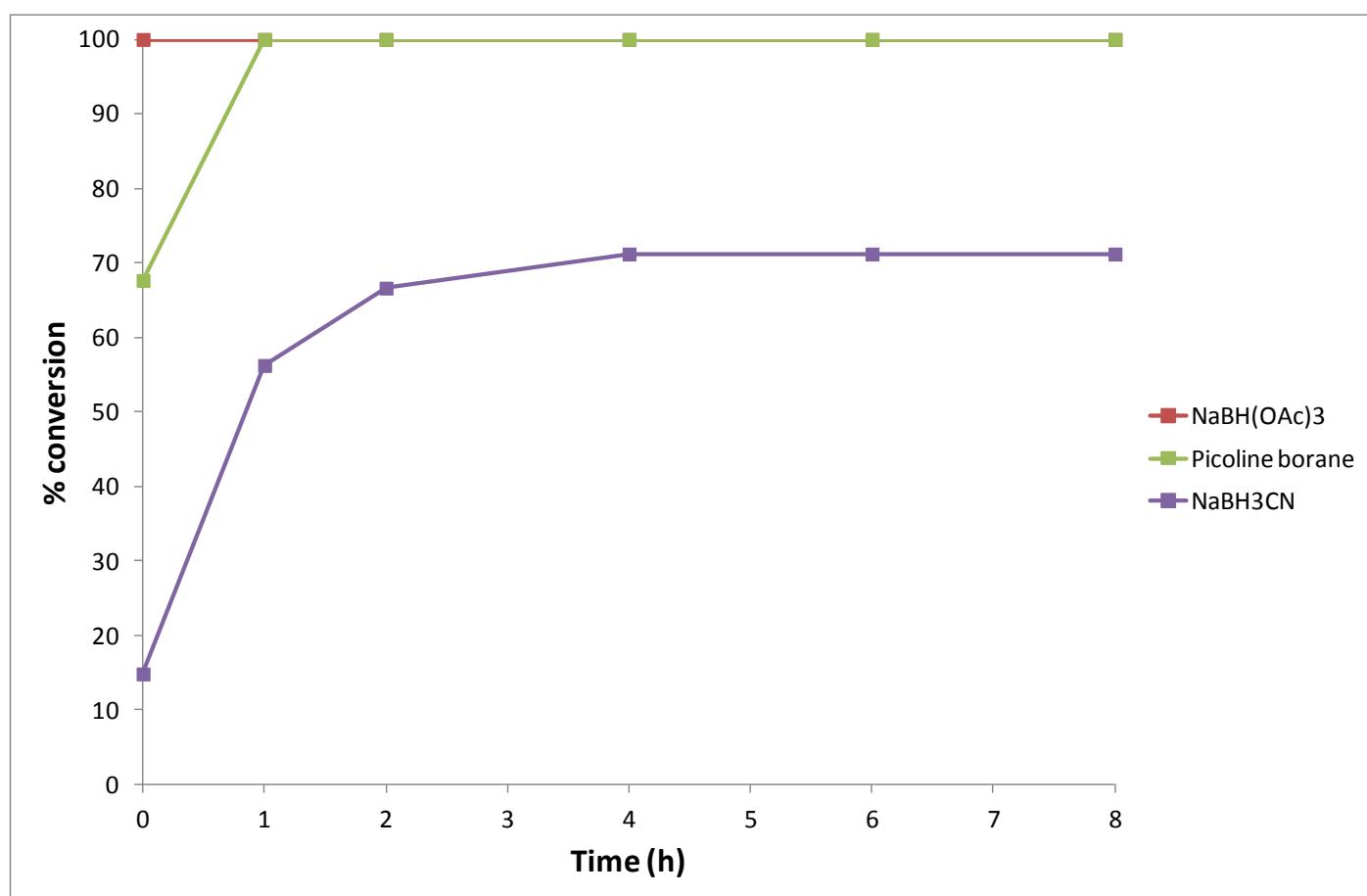
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	28.6	69.3	51.9
1	100.0	100.0	82.9
2	100.0	100.0	89.4
4	100.0	100.0	100.0
6	100.0	100.0	100.0
8	100.0	100.0	100.0
24	100.0	100.0	100.0

Reaction 12: DMC



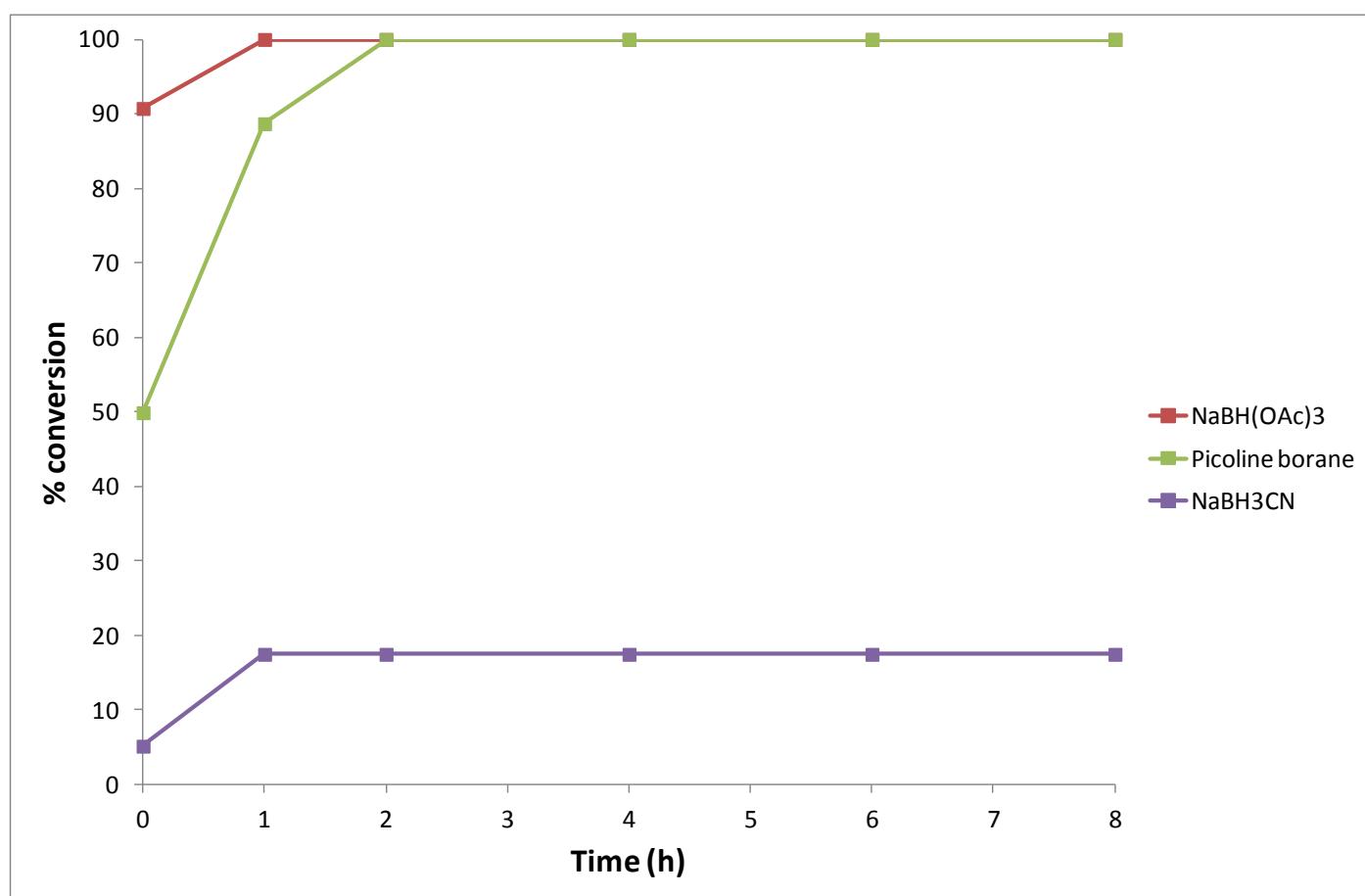
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	72.4	43.0	3.8
1	86.7	100.0	9.0
2	97.3	100.0	9.3
4	100.0	100.0	9.7
6	100.0	100.0	11.2
8	100.0	100.0	12.2
24	100.0	100.0	12.7

Reaction 12: DMF



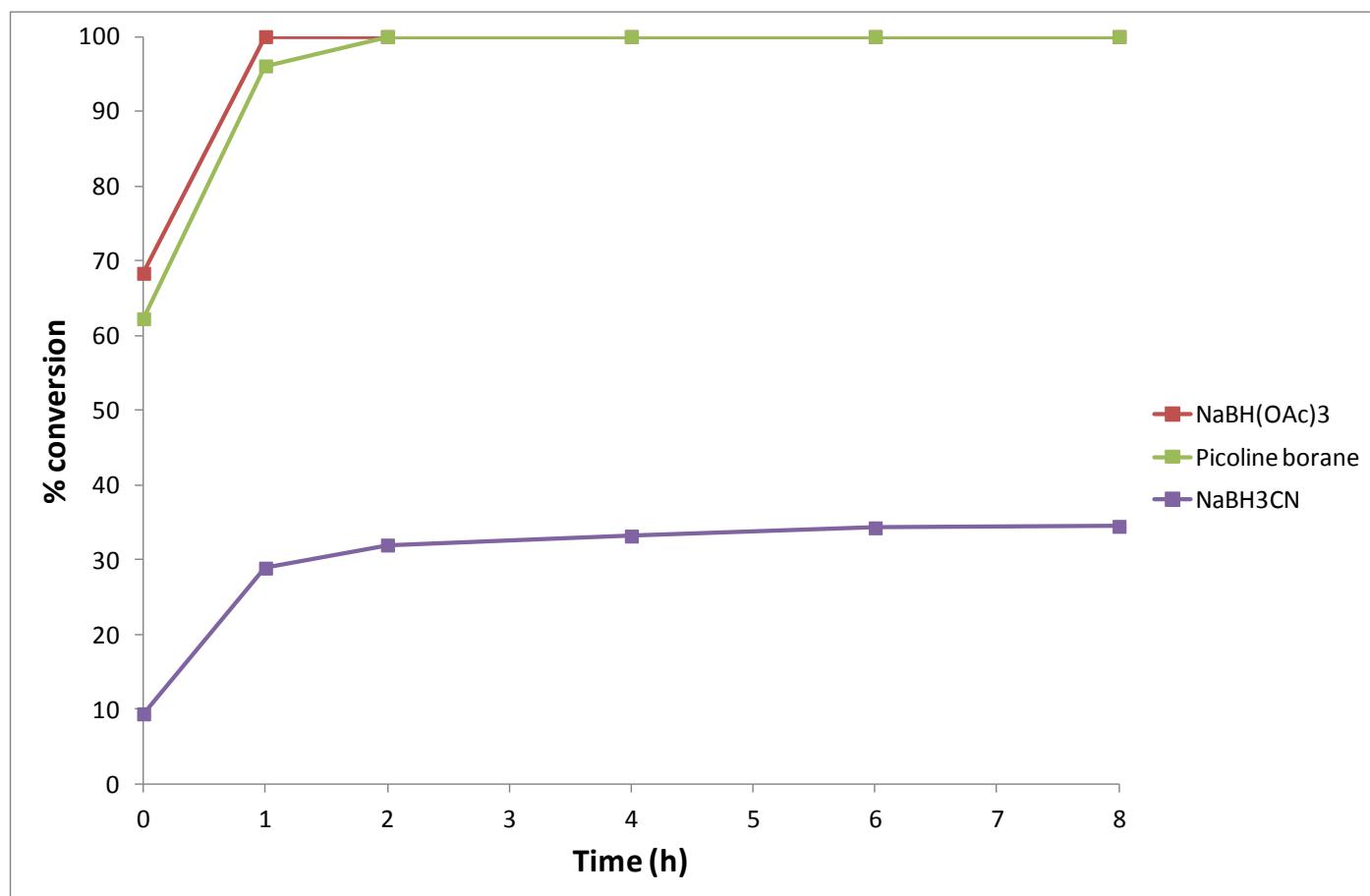
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	100.0	67.6	14.9
1	100.0	100.0	56.3
2	100.0	100.0	66.6
4	100.0	100.0	71.2
6	100.0	100.0	71.2
8	100.0	100.0	71.2
24	100.0	100.0	71.2

Reaction 12: EtOAc



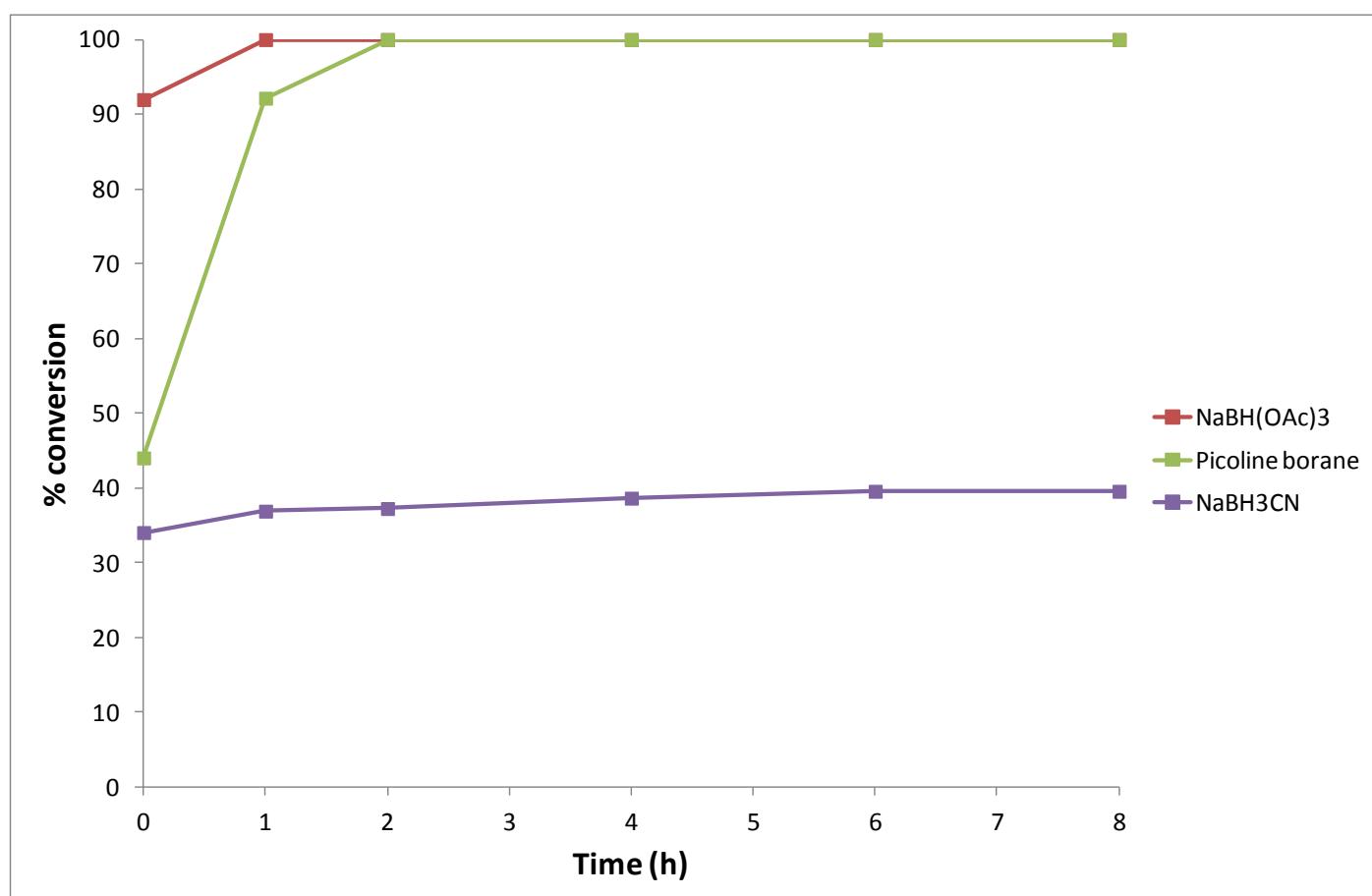
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	90.7	49.9	5.2
1	100.0	88.7	17.5
2	100.0	100.0	17.5
4	100.0	100.0	17.5
6	100.0	100.0	17.5
8	100.0	100.0	17.5
24	100.0	100.0	17.5

Reaction 12: IPA



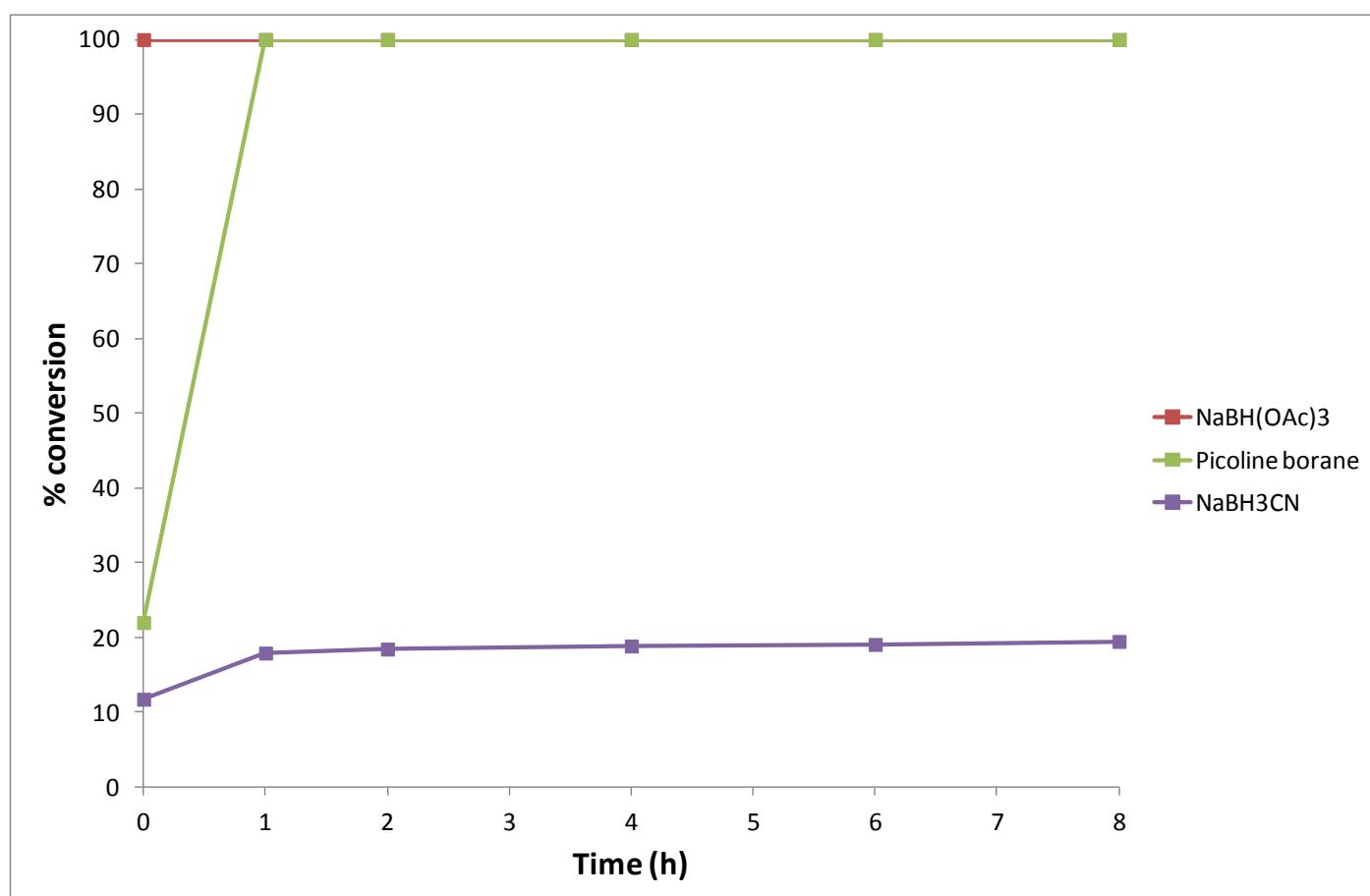
Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	68.3	62.3	9.4
1	100.0	96.1	28.9
2	100.0	100.0	32.0
4	100.0	100.0	33.2
6	100.0	100.0	34.3
8	100.0	100.0	34.5
24	100.0	100.0	37.1

Reaction 12: 2-MeTHF



Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	92.0	44.0	34.1
1	100.0	92.2	36.9
2	100.0	100.0	37.3
4	100.0	100.0	38.7
6	100.0	100.0	39.6
8	100.0	100.0	39.6
24	100.0	100.0	39.6

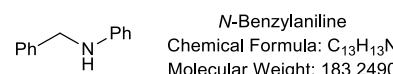
Reaction 12: THF



Time (h) / Reducing agent	NaBH(OAc) ₃	Picoline borane	NaBH ₃ CN
0	100.0	22.0	11.8
1	100.0	100.0	17.9
2	100.0	100.0	18.5
4	100.0	100.0	18.9
6	100.0	100.0	19.1
8	100.0	100.0	19.5
24	100.0	100.0	20.4

3.4 Characterisation Data for Reductive Amination Products

Compound 1: *N*-Benzylaniline



Appearance: White solid.

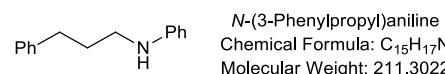
ν_{max} (neat): 3414, 3051, 3024, 2837, 1502, 746 cm⁻¹.

¹H NMR (400 MHz, CDCl₃) δ: 7.39-7.50 (m, 5H), 7.31 (t, 2H, J = 7.6 Hz), 6.86 (t, 1H, J = 7.2 Hz), 6.75 (d, 2H, J = 8.0 Hz), 4.43 (s, 2H), 4.10 (br s, 1H).

¹³C NMR (100 MHz, CDCl₃) δ: 148.4, 139.7, 129.5, 128.9, 127.8, 127.5, 117.8, 113.1, 48.5.

HRMS (C₁₃H₁₃N) [M+H⁺] requires 184.1121, found [M+H⁺] 184.1119.

Compound 2: *N*-(3-Phenylpropyl)aniline



Appearance: Yellow oil.

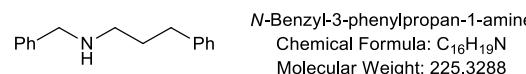
ν_{max} (neat): 3408, 3022, 2927, 2858, 1598, 1504, 742 cm⁻¹.

¹H NMR (400 MHz, CDCl₃) δ: 7.31-7.35 (m, 2H), 7.17-7.25 (m, 5H), 6.72 (tt, 1H, J = 7.2, 1.2 Hz), 6.59-6.62 (m, 2H), 3.64 (br s, 1H), 3.18 (t, 2H, J = 7.2 Hz), 2.77 (t, 2H, J = 7.6 Hz), 1.95-2.02 (m, 2H).

¹³C NMR (100 MHz, CDCl₃) δ: 148.7, 142.0, 129.5, 128.8, 128.7, 126.3, 117.5, 113.1, 43.7, 33.7, 31.4.

HRMS (C₁₅H₁₇N) [M+H⁺] requires 212.1434, found [M+H⁺] 212.1434.

Compound 3: *N*-Benzyl-3-phenylpropan-1-amine



Appearance: Colourless oil.

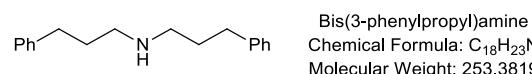
ν_{max} (neat): 3024, 2926, 2814, 2802, 1494, 1452, 731 cm⁻¹.

¹H NMR (400 MHz, CDCl₃) δ: 7.24-7.34 (m, 7H), 7.18-7.21 (m, 3H), 3.80 (s, 2H), 2.69 (m, 4H), 1.83-1.90 (m, 2H), 1.59 (br s, 1H).

¹³C NMR (100 MHz, CDCl₃) δ: 142.5, 140.8, 128.7, 128.7, 128.7, 128.4, 127.3, 126.1, 54.3, 49.2, 33.9, 32.0.

HRMS (C₁₆H₁₉N) [M+H⁺] requires 226.1590, [M+H⁺] found 226.1591.

Compound 4: Bis(3-phenylpropyl)amine



Appearance: Colourless oil.

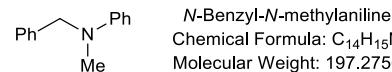
ν_{max} (neat): 2924, 2854, 1496, 1168, 742 cm^{-1} .

^1H NMR (400 MHz, CDCl_3) δ : 8.19 (br s, 1H), 7.26-7.31 (m, 4H), 7.18-7.22 (m, 2H), 7.11-7.13 (m, 4H), 2.82 (4H, J = 8.0 Hz), 2.58 (t, 4H, J = 7.6 Hz), 1.95 (pent, 4H, J = 7.6 Hz).

^{13}C NMR (100 MHz, CDCl_3) δ : 140.4, 128.9, 128.5, 126.6, 47.9, 32.9, 28.3.

HRMS ($\text{C}_{18}\text{H}_{23}\text{N}$) [$\text{M}+\text{H}^+$] requires 254.1903, found $[\text{M}+\text{H}^+]$ 254.1906.

Compound 5: *N*-Benzyl-*N*-methylaniline



Appearance: Yellow oil.

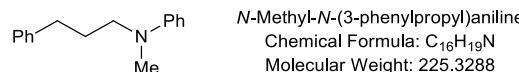
ν_{max} (neat): 3024, 2881, 2804, 1597, 1573, 943, 746 cm^{-1} .

^1H NMR (400 MHz, CDCl_3) δ : 7.44-7.47 (m, 2H), 7.35-7.39 (m, 5H), 6.85-6.92 (m, 3H), 4.67 (s, 2H), 3.15 (s, 3H).

^{13}C NMR (100 MHz, CDCl_3) δ : 150.1, 139.3, 129.5, 128.9, 127.2, 127.1, 116.9, 112.7, 56.9, 38.8.

HRMS ($\text{C}_{14}\text{H}_{15}\text{N}$) [$\text{M}+\text{H}^+$] requires 198.1277, $[\text{M}+\text{H}^+]$ found 198.1277.

Compound 6: *N*-Methyl-*N*-(3-phenylpropyl)aniline



Appearance: Yellow oil.

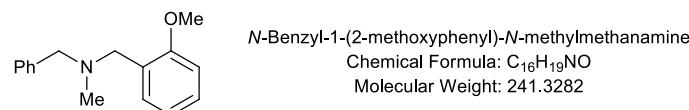
ν_{max} (neat): 3024, 2935, 2862, 1597, 1573, 989, 742 cm^{-1} .

^1H NMR (500 MHz, CDCl_3) δ : 7.31 (t, 2H, J = 6.0 Hz), 7.20-7.25 (m, 5H), 6.68-6.71 (m, 3H), 3.36 (t, 2H, J = 5.6 Hz), 2.94 (s, 3H), 2.68 (t, 2H, J = 6.4 Hz), 1.94 (pent, 2H, J = 6.0 Hz).

^{13}C NMR (100 MHz, CDCl_3) δ : 149.7, 142.2, 129.5, 128.7, 128.7, 126.2, 116.4, 112.6, 52.6, 38.6, 33.7, 28.5.

HRMS ($\text{C}_{16}\text{H}_{19}\text{N}$) [$\text{M}+\text{H}^+$] requires 226.1590, $[\text{M}+\text{H}^+]$ found 226.1590.

Compound 7: *N*-Benzyl-1-(2-methoxyphenyl)-*N*-methylmethanamine



Appearance: Yellow oil.

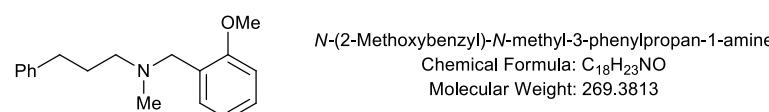
ν_{max} (neat): 3026, 2933, 2833, 1490, 1238, 1026 cm^{-1} .

^1H NMR (400 MHz, CDCl_3) δ : 7.45 (dd, 1H, J = 7.2, 1.6 Hz), 7.39 (d, 2H, J = 7.2 Hz), 7.31-7.35 (m, 2H), 7.22-7.27 (m, 2H), 6.96 (td, 1H, J = 7.2, 0.8 Hz), 6.87 (d, 1H, J = 8.4 Hz), 3.82 (s, 3H), 3.60 (br s, 4H), 2.24 (s, 3H).

^{13}C NMR (100 MHz, CDCl_3) δ : 158.2, 130.7, 129.4, 128.5, 128.2, 127.2, 120.7, 110.8, 62.6, 55.7, 55.5, 42.7. Two aromatic C coincident with other signals.

HRMS ($\text{C}_{16}\text{H}_{19}\text{NO}$) [$\text{M}+\text{H}^+$] requires 242.1539, $[\text{M}+\text{H}^+]$ found 242.1540.

Compound 8: N-(2-Methoxybenzyl)-N-methyl-3-phenylpropan-1-amine



Appearance: Colourless oil.

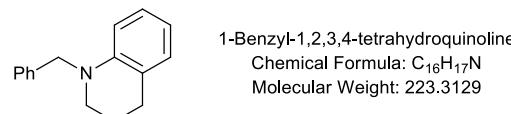
ν_{max} (neat): 2935, 2835, 2787, 1490, 1454, 1238, 1028 cm⁻¹.

¹H NMR (400 MHz, CDCl₃) δ: 7.34 (dd, 1H, J = 7.2, 1.6 Hz), 7.25-7.30 (m, 3H), 7.17-7.23 (m, 3H), 6.94 (td, 1H, J = 7.2, 0.8 Hz), 6.88 (d, 1H, J = 8.0 Hz), 3.83 (s, 3H), 3.54 (s, 2H), 2.67 (t, 2H, J = 7.6 Hz), 2.48 (t, 2H, J = 7.2 Hz), 2.24 (s, 3H), 1.89 (pent, 2H, J = 7.2 Hz).

¹³C NMR (100 MHz, CDCl₃) δ: 169.0, 158.3, 142.4, 131.6, 128.9, 128.8, 128.7, 126.1, 120.8, 110.8, 56.9, 55.7, 5.1, 42.0, 33.9, 28.8.

HRMS (C₁₈H₂₃NO) [M+H⁺] requires 270.1852, [M+H⁺] found 270.1855.

Compound 9: 1-Benzyl-1,2,3,4-tetrahydroquinoline



Appearance: Pink oil.

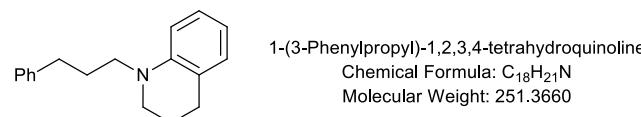
ν_{max} (neat): 3022, 2924, 2839, 1504, 1494, 1344, 986 cm⁻¹.

¹H NMR (400 MHz, CDCl₃) δ: 7.23-7.35 (m, 5H), 6.97-7.00 (m, 2H), 6.59 (t, 1H, J = 7.6 Hz), 6.52 (d, 1H, J = 8.4 Hz), 4.50 (s, 2H), 3.38 (t, 2H, J = 6.0 Hz), 2.84 (t, 2H, J = 6.4 Hz), 2.03 (pent, 2H, J = 5.6 Hz).

¹³C NMR (100 MHz, CDCl₃) δ: 145.9, 139.2, 129.3, 128.9, 127.5, 127.1, 126.9, 122.6, 116.2, 111.3, 55.5, 50.2, 28.5, 22.7.

HRMS (C₁₆H₁₇N) [M+H⁺] requires 224.1434, [M+H⁺] found 224.1434.

Compound 10: 1-(3-Phenylpropyl)-1,2,3,4-tetrahydroquinoline



Appearance: Orange crystals.

ν_{max} (neat): 3408, 3024, 2926, 2839, 1494, 1307 cm⁻¹.

¹H NMR (400 MHz, CDCl₃) δ: 7.29-7.33 (m, 2H), 7.20-7.24 (m, 3H), 6.96-7.00 (m, 2H), 6.65 (t, 1H, J = 7.2 Hz), 6.53 (d, 1H, J = 7.6 Hz), 3.41 (t, 2H, J = 6.8 Hz), 3.32 (t, 2H, J = 5.6 Hz), 2.76-2.81 (m, 4H), 2.15-2.22 (m, 2H), 1.94-2.00 (m, 2H).

¹³C NMR (100 MHz, CDCl₃) δ: 145.6, 142.2, 129.5, 128.7, 128.7, 127.4, 126.2, 122.6, 115.7, 110.8, 51.3, 49.8, 33.7, 28.5, 28.0, 22.6.

HRMS (C₁₈H₂₁N) [M+H⁺] requires 252.1702, [M+H⁺] found 252.1749.

Compound 11: 1,4-Dibenzylpiperidine



Appearance: White solid.

ν_{max} (neat): 3061, 3024, 927, 2906, 2794, 2746, 1573, 1560, 970, 736 cm⁻¹.

¹H NMR (400 MHz, CDCl₃) δ: 7.21-7.33 (m, 7H), 7.12-7.16 (m, 1H), 7.08 (d, 2H, J = 6.8 Hz), 3.55 (s, 2H), 2.92 (d, 2H, J = 11.6 Hz), 2.49 (d, 2H, J = 6.8 Hz), 1.96 (t, 2H, J = 10.0 Hz, 1.59 (d, 2H, J = 13.2 Hz), 1.39-1.49 (m, 1H), 1.33-1.37 (m, 2H).

¹³C NMR (100 MHz, CDCl₃) δ: 141.1, 138.7, 129.6, 129.5, 128.5, 127.2, 126.1, 63.7, 54.1, 43.6, 38.2, 32.5. One aromatic C coincident with another signal.

HRMS (C₁₉H₂₃N) [M+H⁺] requires 266.1903, [M+H⁺] found 266.1905.

Compound 12: 4-Benzyl-1-(3-phenylpropyl)piperidine



Appearance: Colourless oil.

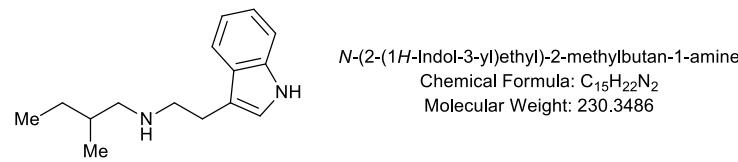
ν_{max} (neat): 3024, 2914, 2800, 2765, 1494, 1452, 742 cm⁻¹.

¹H NMR (400 MHz, CDCl₃) δ: 7.27-7.31 (m, 4H), 7.15-7.22 (m, 6H), 2.92 (d, 2H, J = 11.6 Hz), 2.64 (t, 2H, J = 7.6 Hz), 2.50 (d, 2H, J = 6.8 Hz), 2.35 (t, 2H, J = 7.6 Hz), 1.80-1.90 (m, 4H), 1.65 (d, 2H, J = 13.2 Hz), 1.51-1.55 (m, 1H), 1.28-1.36 (m, 2H).

¹³C NMR (100 MHz, CDCl₃) δ: 142.6, 141.1, 129.5, 128.7, 128.6, 128.5, 126.1, 126.0, 58.8, 54.3, 43.6, 38.3, 34.2, 32.6, 29.1.

HRMS (C₂₁H₂₇N) [M+H⁺] requires 294.2216, [M+H⁺] found 294.2219.

Compound 13: N-(2-(1*H*-Indol-3-yl)ethyl)-2-methylbutan-1-amine



Appearance: Orange oil.

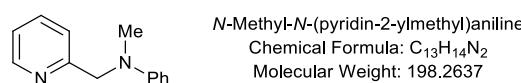
ν_{max} (neat): 3410, 2956, 2918, 2850, 2362, 1454 cm⁻¹.

¹H NMR (400 MHz, CDCl₃) δ: 8.05 (br s, 1H), 7.65 (d, 1H, J = 6.4 Hz), 7.37 (d, 1H, J = 6.8 Hz), 7.19-7.22 (m, 1H), 7.11-7.14 (m, 1H), 7.06 (d, 1H, J = 1.2 Hz), 2.97-3.05 (m, 4H), 2.59-2.63 (m, 1H), 2.43-2.47 (m, 1H), 2.09 (br s, 1H), 1.55-1.59 (m, 1H), 1.36-1.40 (m, 1H), 1.09-1.15 (m, 1H), 0.85-0.88 (m, 6H).

¹³C NMR (100 MHz, CDCl₃) δ: 136.8, 127.8, 122.4, 122.2, 119.7, 119.3, 114.3, 111.5, 56.1, 50.4, 34.9, 27.9, 25.8, 18.0, 11.6.

HRMS (C₁₅H₂₂N₂) [M+H⁺] requires 231.1856, [M+H⁺] found 231.1856.

Compound 14: *N*-Methyl-*N*-(pyridin-2-ylmethyl)aniline



Appearance: Off-white solid.

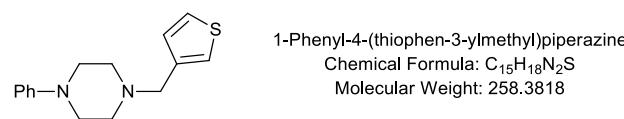
ν_{max} (neat): 2980, 2920, 2363, 1598, 1585, 1504 cm⁻¹.

¹H NMR (400 MHz, CDCl₃) δ: 8.60 (d, 1H, J = 4.0 Hz), 7.62 (dt, 1H, J = 7.6, 1.6 Hz), 7.17-7.25 (m, 4H), 6.72-6.75 (m, 3H), 4.68 (s, 2H), 3.13 (s, 3H).

¹³C NMR (100 MHz, CDCl₃) δ: 159.7, 149.7, 149.5, 137.3, 129.6, 122.3, 121.2, 117.1, 112.5, 59.1, 39.4.

HRMS (C₁₃H₁₄N₂) [M+H⁺] requires 199.1230, [M+H⁺] found 199.1224.

Compound 15: 1-Phenyl-4-(thiophen-3-ylmethyl)piperazine



Appearance: White solid.

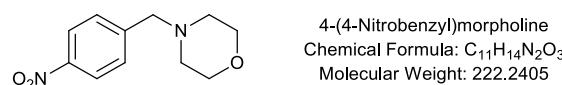
ν_{max} (neat): 3082, 2933, 2845, 2802, 2362, 1597, 1502, 1334, 1238 cm⁻¹.

¹H NMR (400 MHz, CDCl₃) δ: 7.25-7.31 (m, 3H), 7.17 (d, 1H, J = 2.0 Hz), 7.11 (dd, 1H, J = 3.6, 1.2 Hz), 6.92-6.95 (m, 2H), 6.86 (tt, 1H, J = 7.2, 0.8 Hz), 3.62 (s, 2H), 3.22 (t, 4H, J = 5.2 Hz), 2.63 (t, 4H, J = 5.2 Hz).

¹³C NMR (100 MHz, CDCl₃) δ: 151.7, 139.2, 129.4, 128.9, 125.8, 123.3, 120.0, 116.4, 58.0, 53.4, 49.5.

HRMS (C₁₅H₁₈N₂S) [M+H⁺] requires 259.1263, [M+H⁺] found 259.1266.

Compound 16: 4-(4-Nitrobenzyl)morpholine



Appearance: Yellow solid.

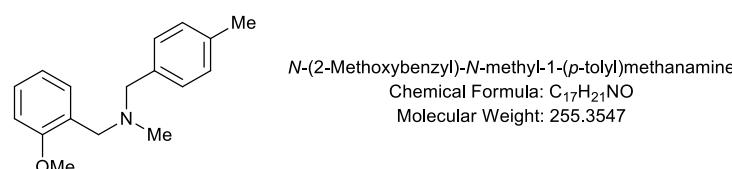
ν_{max} (neat): 2960, 2852, 2806, 2362, 1516, 1340, 1114 cm⁻¹.

¹H NMR (400 MHz, CDCl₃) δ: 8.17 (dt, 2H, J = 6.8, 2.0 Hz), 7.52 (d, 2H, J = 8.4 Hz), 3.72 (t, 4H, J = 4.4 Hz), 3.59 (s, 2H), 2.47 (t, 4H, J = 4.8 Hz).

¹³C NMR (100 MHz, CDCl₃) δ: 147.5, 146.1, 129.8, 123.8, 67.2, 62.8, 53.9.

HRMS (C₁₁H₁₄N₂O₃) [M+H⁺] requires 223.1077, [M+H⁺] found 223.1077.

Compound 17: *N*-(3-Methoxybenzyl)-*N*-methyl-1-(*p*-tolyl)methanamine



Appearance: Colourless oil.

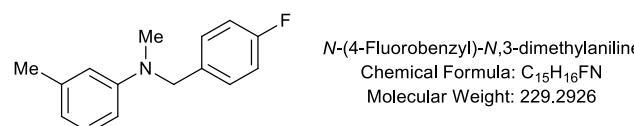
ν_{max} (neat): 2980, 2833, 2787, 2358, 1236, 1029 cm^{-1} .

^1H NMR (400 MHz, CDCl_3) δ : 7.47 (dd, 1H, J = 7.6, 1.6 Hz), 7.30 (d, 2H, J = 7.6 Hz), 7.23-7.27 (m, 1H), 7.16 (d, 2H, J = 7.6 Hz), 6.98 (t, 1H, J = 7.2 Hz), 6.89 (d, 1H, J = 8.0 Hz), 3.84 (s, 3H), 3.66 (s, 2H), 3.58 (s, 2H), 2.37 (s, 3H), 2.24 (s, 3H).

^{13}C NMR (100 MHz, CDCl_3) δ : 158.1, 136.7, 136.6, 130.7, 129.3, 129.1, 128.1, 127.7, 120.6, 110.7, 62.3, 55.7, 55.4, 42.6, 21.4.

HRMS ($\text{C}_{17}\text{H}_{21}\text{NO}$) [$\text{M}+\text{H}^+$] requires 256.1696, [$\text{M}+\text{H}^+$] found 256.1698.

Compound 18: *N*-(4-Fluorobenzyl)-*N*,3-dimethylaniline



Appearance: Yellow oil.

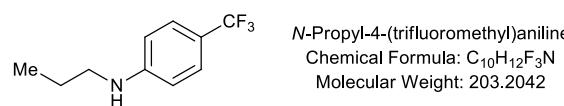
ν_{max} (neat): 3039, 2980, 2891, 2362, 1598, 1492, 1217 cm^{-1} .

^1H NMR (400 MHz, CDCl_3) δ : 7.19-7.23 (m, 2H), 7.11-7.15 (m, 1H), 6.99-7.03 (m, 2H), 6.57-6.59 (m, 3H), 4.49 (s, 2H), 2.98 (s, 3H), 2.32 (s, 3H).

^{13}C NMR (100 MHz, CDCl_3) δ : 162.3 (d, $^1J_{\text{C-F}} = 244.6$ Hz), 150.2, 139.3, 135.1, 129.4, 128.7 (d, $^3J_{\text{C-F}} = 8.0$ Hz), 118.2, 115.7 (d, $^2J_{\text{C-F}} = 21.3$ Hz), 113.6, 110.1, 56.4, 38.7, 22.2.

HRMS ($\text{C}_{15}\text{H}_{16}\text{FN}$) [$\text{M}+\text{H}^+$] requires 230.1340, [$\text{M}+\text{H}^+$] found 230.1339.

Compound 19: *N*-Propyl-4-(trifluoromethyl)aniline



Appearance: Brown oil.

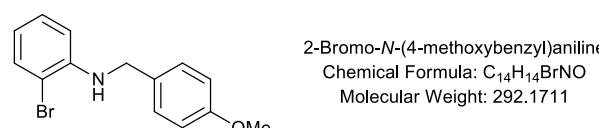
ν_{max} (neat): 3425, 2964, 2877, 1616, 1553, 1317, 1101, 1060 cm^{-1} .

^1H NMR (400 MHz, CDCl_3) δ : 7.40 (d, 2H, J = 8.4 Hz), 6.60 (d, 2H, J = 8.4 Hz), 4.10 (br s, 1H), 3.12 (t, 2H, J = 6.8 Hz), 1.60 (sextet, 2H, J = 7.2 Hz), 1.02 (t, 2H, J = 7.6 Hz).

^{13}C NMR (100 MHz, CDCl_3) δ : 151.07, 126.9 (q, $^3J_{\text{C-F}} = 3.6$ Hz), 125.4 (q, $^1J_{\text{C-F}} = 270$ Hz), 119.0 (q, $^2J_{\text{C-F}} = 32.9$ Hz), 112.2, 45.8, 22.8, 11.9.

HRMS ($\text{C}_{10}\text{H}_{12}\text{F}_3\text{N}$) [$\text{M}+\text{H}^+$] requires 204.0995, [$\text{M}+\text{H}^+$] found 204.0996.

Compound 20: 2-Bromo-*N*-(4-methoxybenzyl)aniline



Appearance: White crystalline solid.

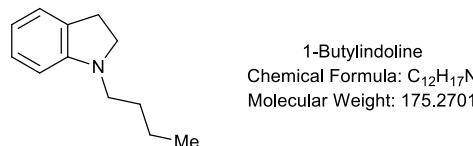
ν_{max} (neat): 3450, 2980, 2870, 2357, 1583, 1083 cm^{-1} .

^1H NMR (400 MHz, CDCl_3) δ : 7.44 (dd, 1H, J = 8.0, 1.6 Hz), 7.30 (d, 2H, J = 8.8 Hz), 7.13-7.17 (m, 1H), 6.90 (dt, 2H, J = 8.8, 2.8 Hz), 6.64, (dd, 1H, J = 8.0, 1.2 Hz), 6.59 (td, 1H, J = 7.6, 1.6 Hz), 4.71 (br s, 1H), 4.34 (s, 2H), 3.82 (s, 3H).

^{13}C NMR (100 MHz, CDCl_3) δ : 159.3, 145.2, 132.7, 130.9, 128.9, 128.8, 118.3, 114.5, 112.0, 110.0, 55.6, 47.9.

HRMS ($\text{C}_{14}\text{H}_{14}\text{BrNO}$) $[\text{M}+\text{H}^+]$ requires 292.0332, $[\text{M}+\text{H}^+]$ found 292.0324.

Compound 21: 1-Butylindoline



Appearance: Clear oil.

ν_{max} (neat): 2954, 2927, 1608, 1487, 1458, 1265 cm^{-1} .

^1H NMR (400 MHz, CDCl_3) δ : 7.10-7.13 (m, 2H), 6.69 (td, 1H, J = 7.2, 0.8 Hz), 6.52 (d, 1H, J = 7.6 Hz), 3.39 (t, 2H, J = 8.4 Hz), 3.11 (t, 2H, J = 7.6 Hz), 3.01 (t, 2H, J = 8.4 Hz), 1.62-1.69 (m, 2H), 1.48 (sextet, 2H, J = 7.4 Hz), 1.03 (t, 3H, J = 7.6 Hz).

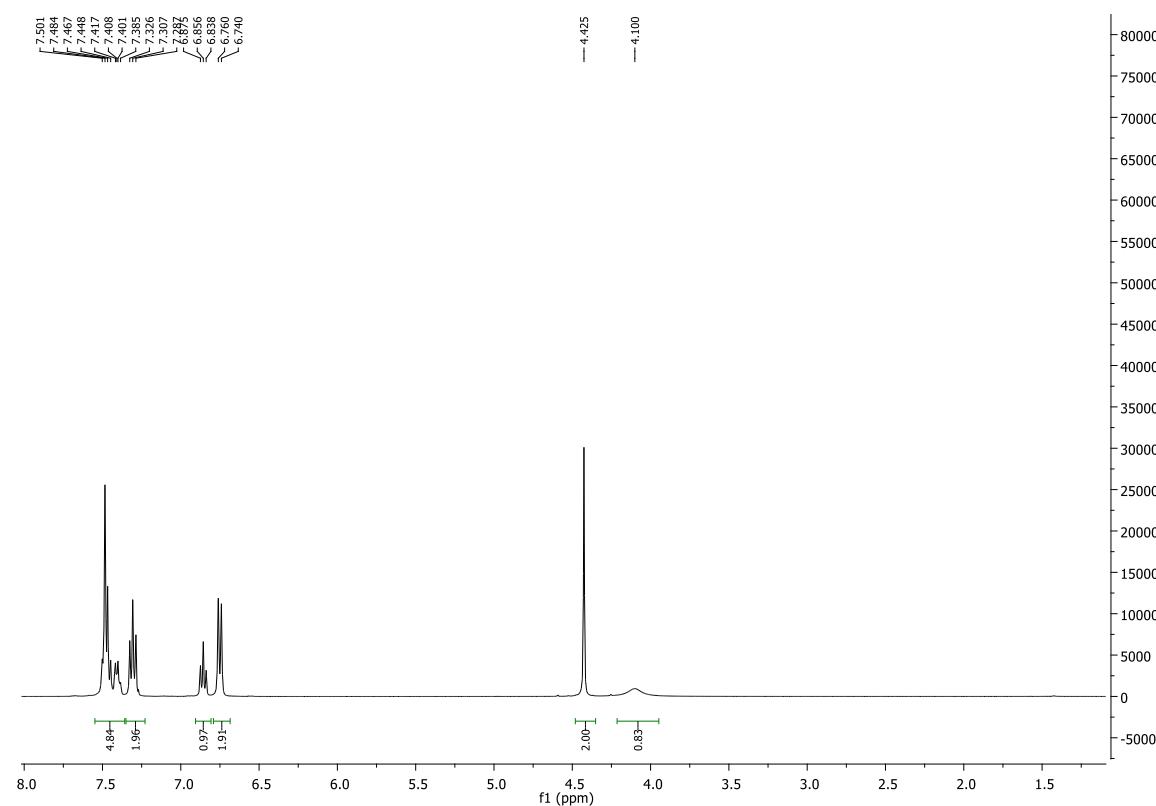
^{13}C NMR (100 MHz, CDCl_3) δ : 153.1, 130.3, 127.6, 125.6, 117.5, 107.1, 53.4, 49.3, 29.8, 28.9, 20.7, 14.3.

HRMS ($\text{C}_{12}\text{H}_{17}\text{N}$) $[\text{M}+\text{H}^+]$ requires 176.1434, $[\text{M}+\text{H}^+]$ found 176.1432.

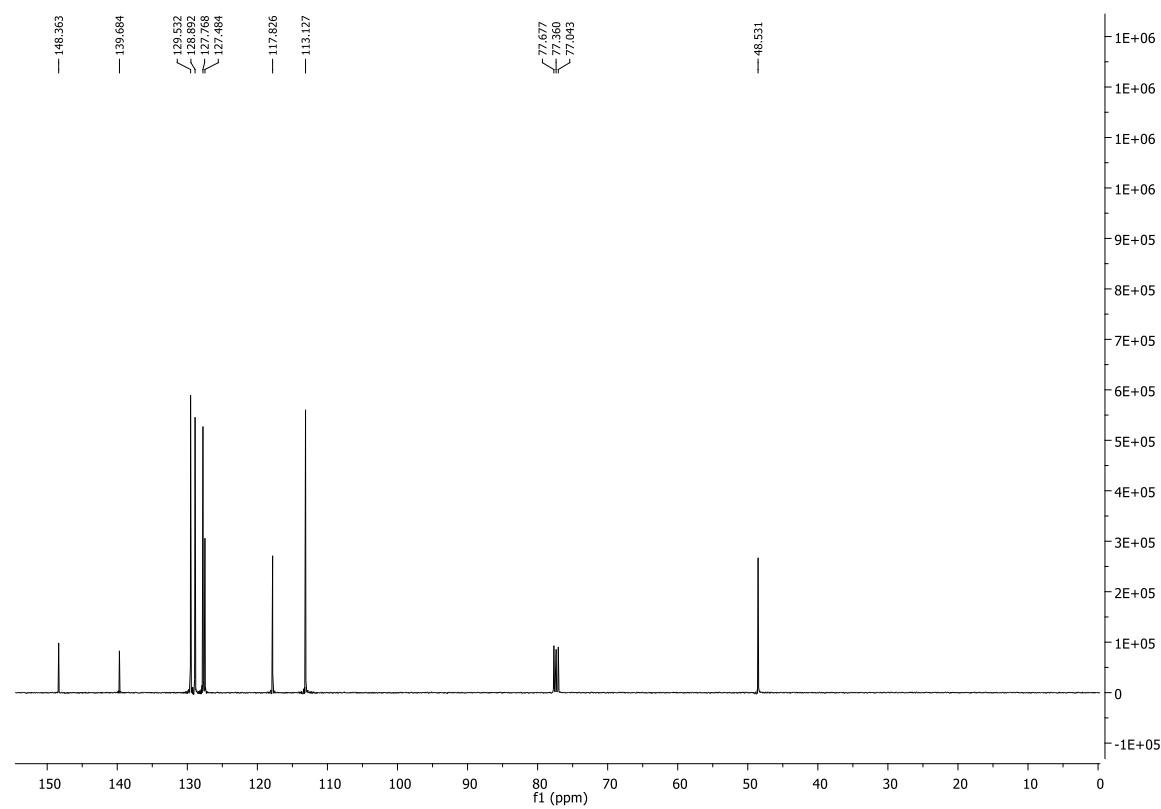
3.5 IR, ^1H NMR, ^{13}C NMR, HRMS, IR and HPLC Spectra for Compounds 1-21

Compound 1: *N*-Benzylaniline

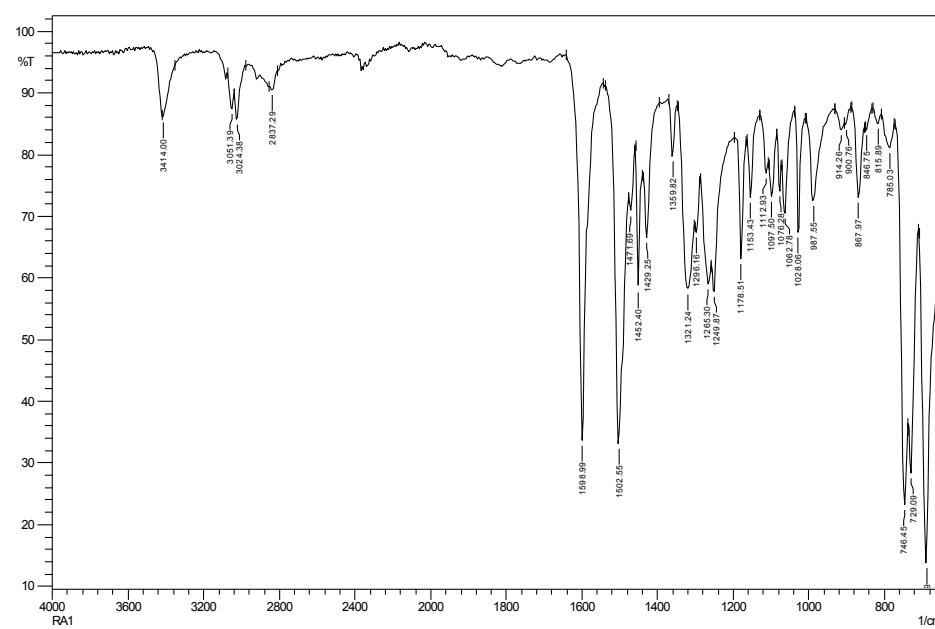
^1H NMR:



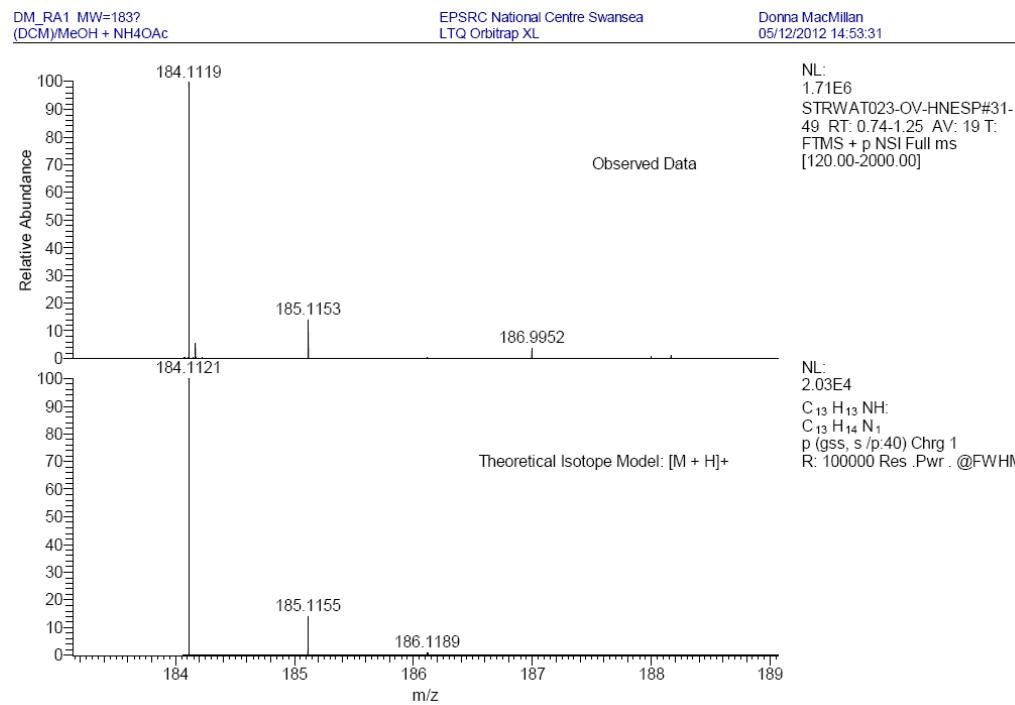
^{13}C NMR:



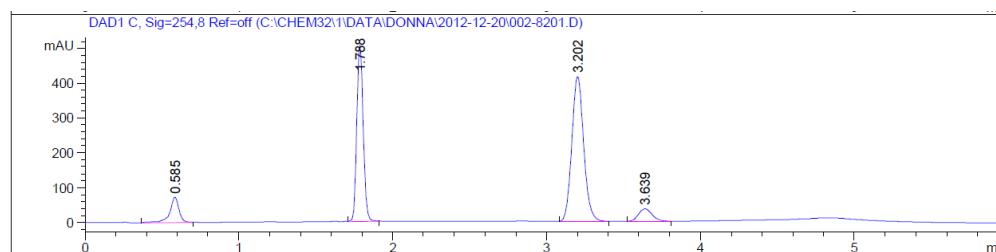
FTIR:



HRMS:



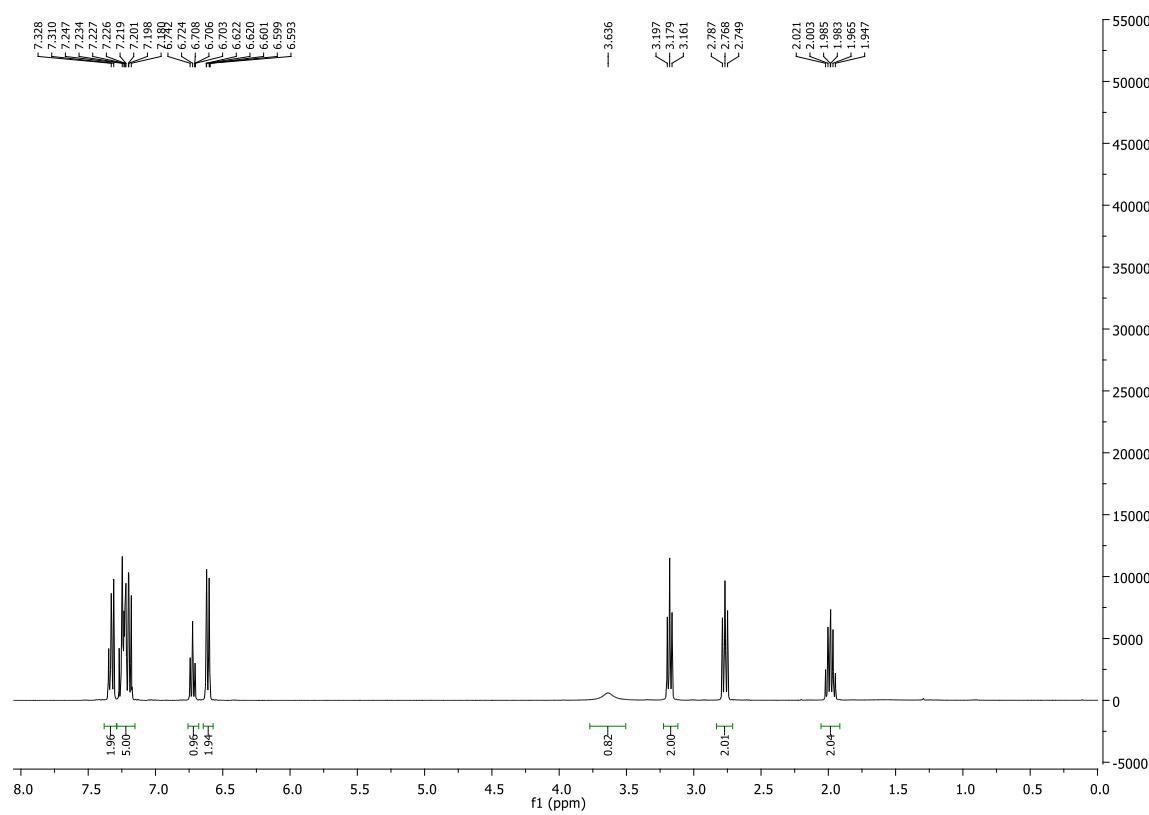
HPLC assay:



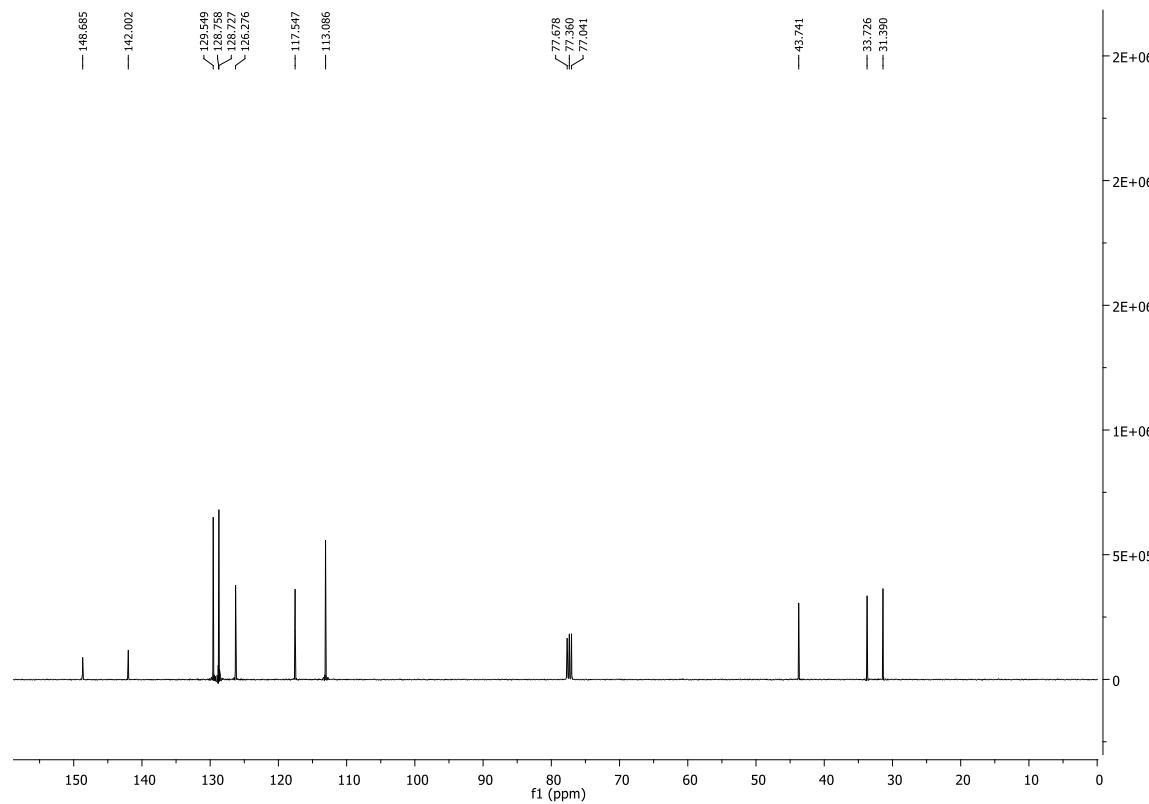
Substrate	R _t (min)
Compound 1: N-Benzylamine	3.20
Iodobenzene (standard)	3.64

Compound 2: N-(3-Phenylpropyl)aniline

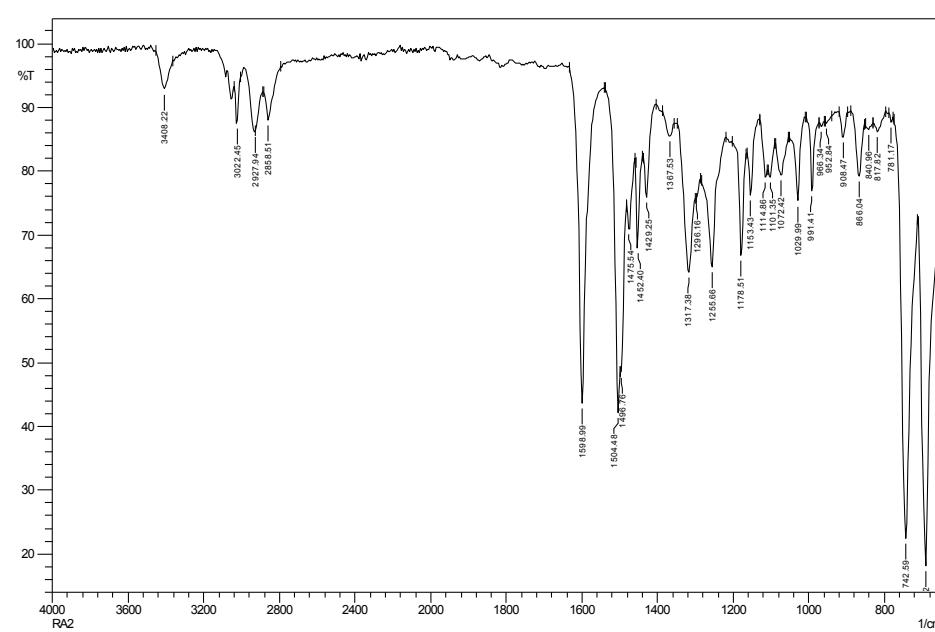
^1H NMR:



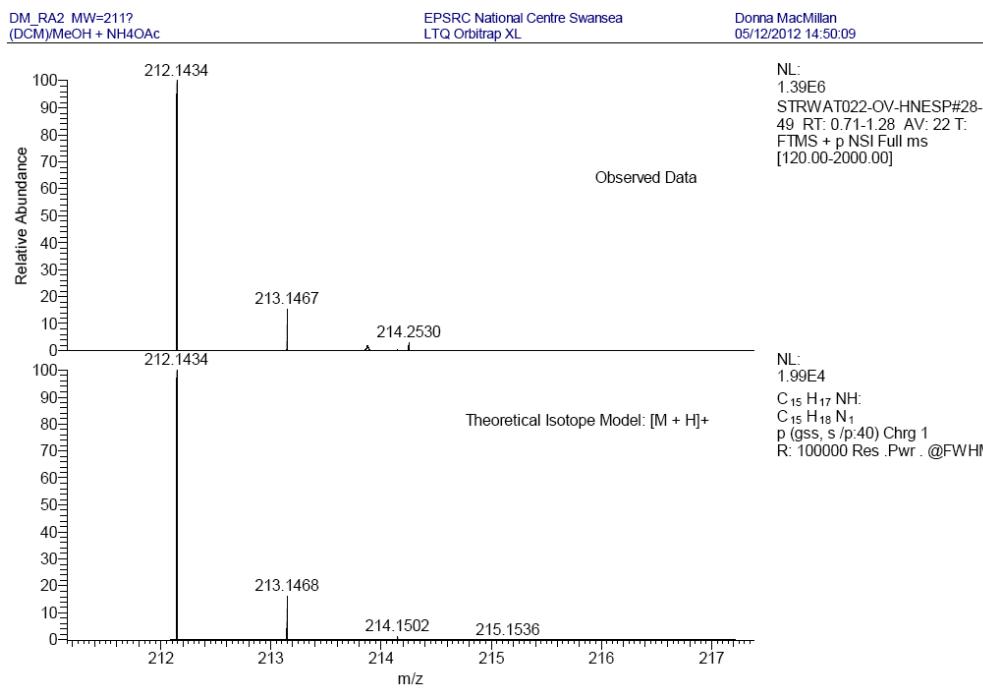
^{13}C NMR:



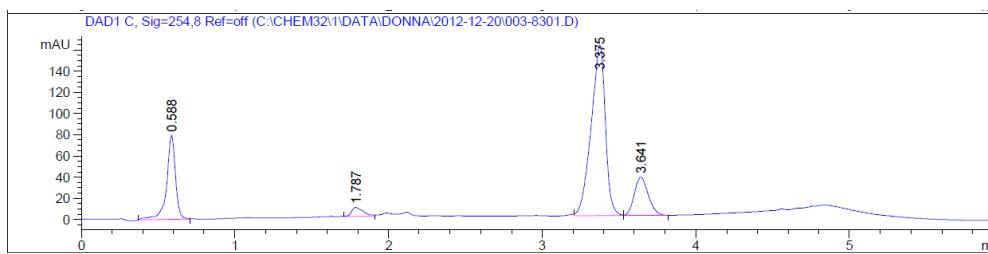
FTIR:



HRMS:



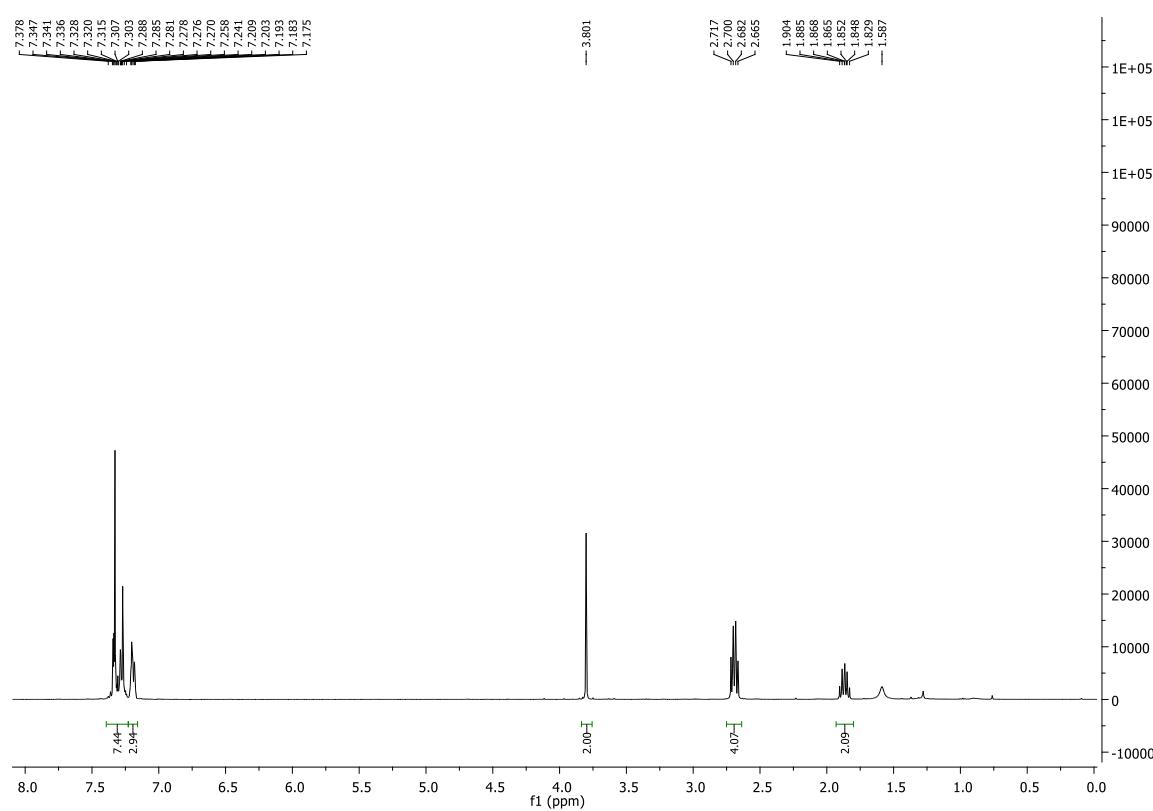
HPLC assay:



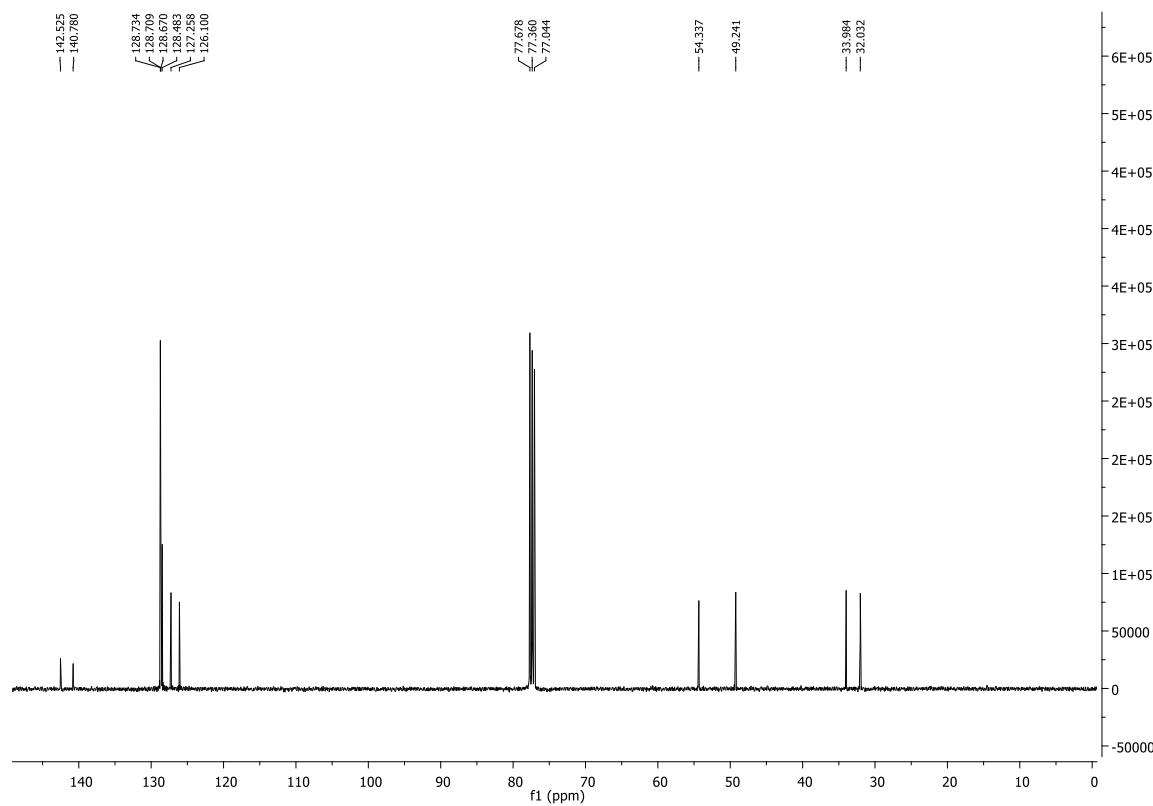
Substrate	R _t (min)
Compound 2: N-(3-phenylpropyl)aniline	3.38
Iodobenzene (standard)	3.64

Compound 3: N-Benzyl-3-phenylpropan-1-amine

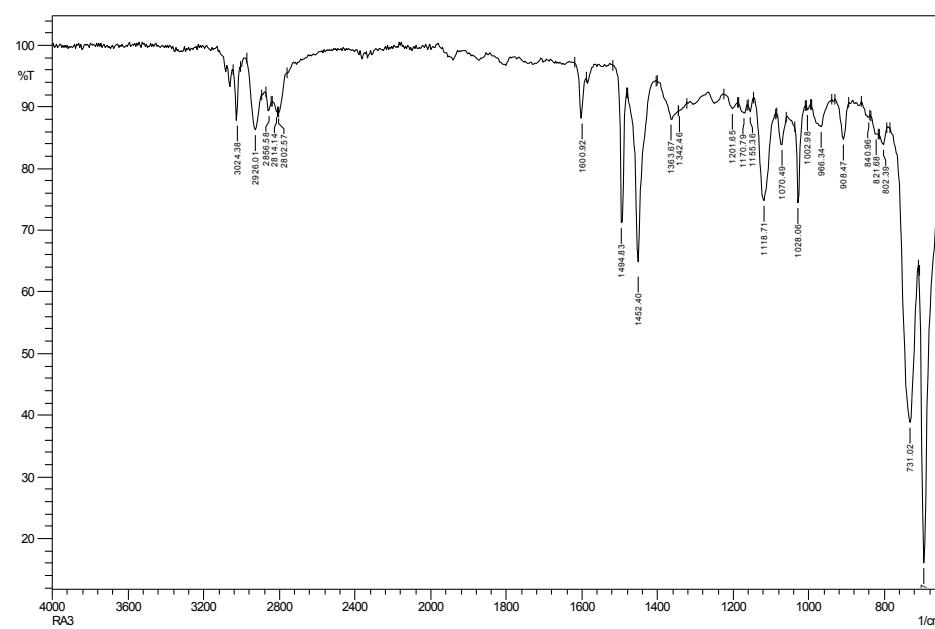
^1H NMR:



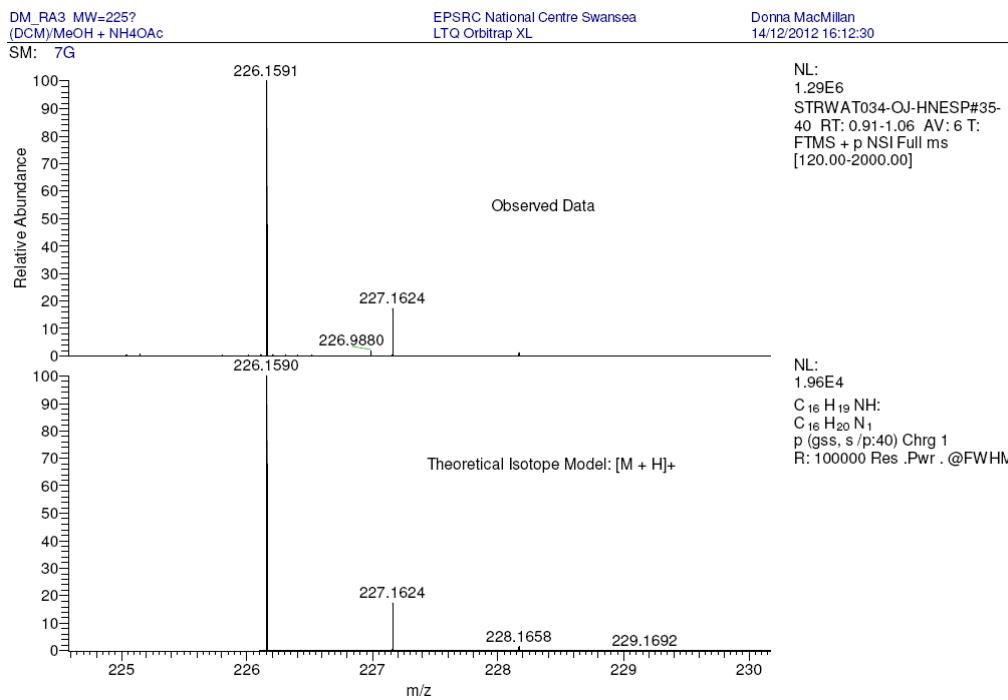
^{13}C NMR:



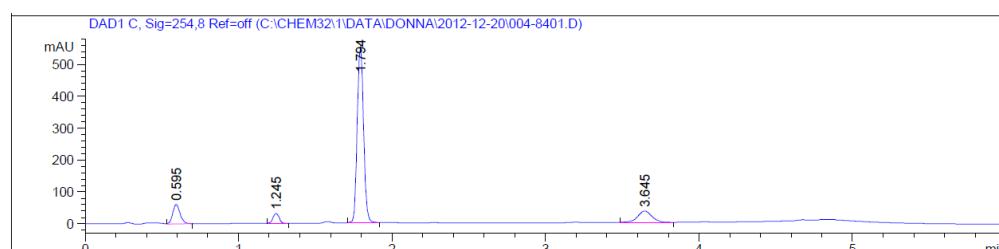
FTIR:



HRMS:



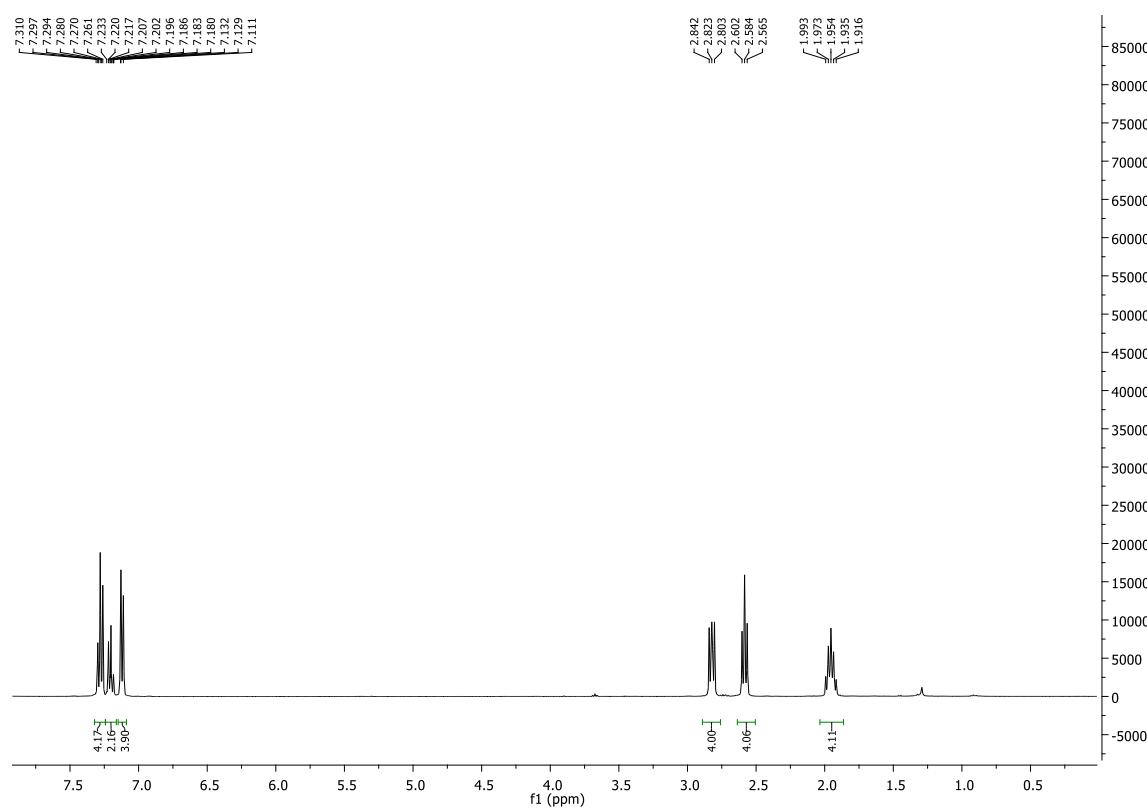
HPLC assay:



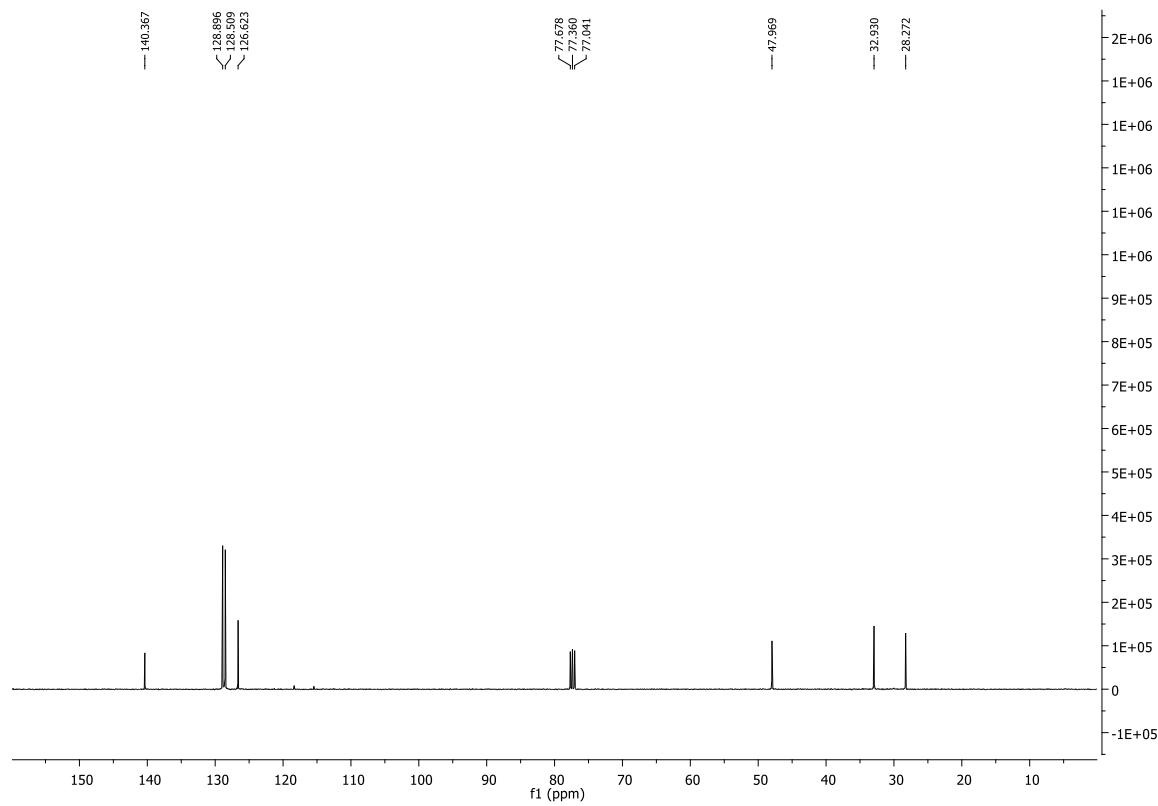
Substrate	R _t (min)
Compound 3: N-benzyl-3-phenylpropan-1-amine	1.25
Iodobenzene (standard)	3.64

Compound 4: Bis(3-phenylpropyl)amine

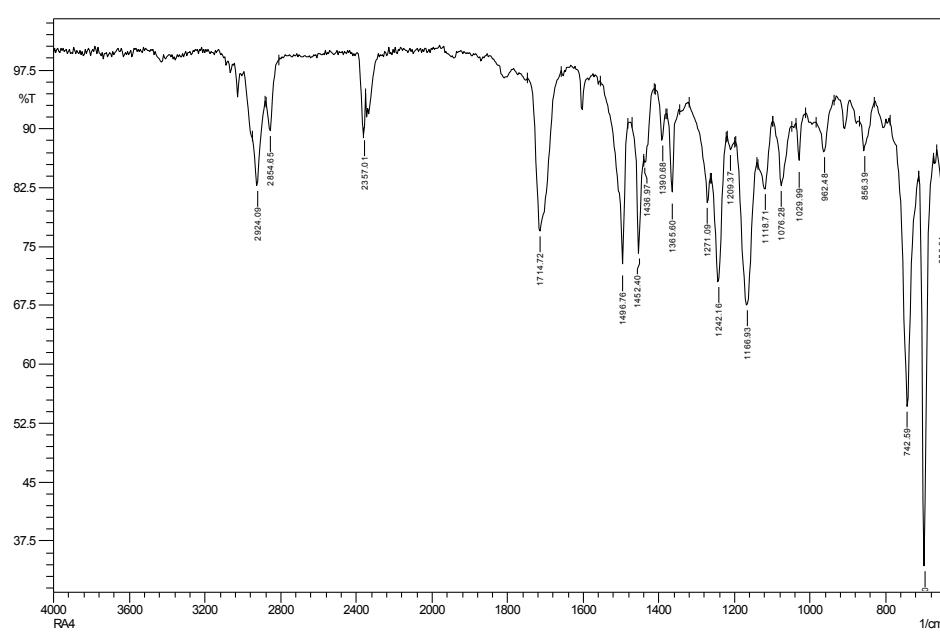
^1H NMR:



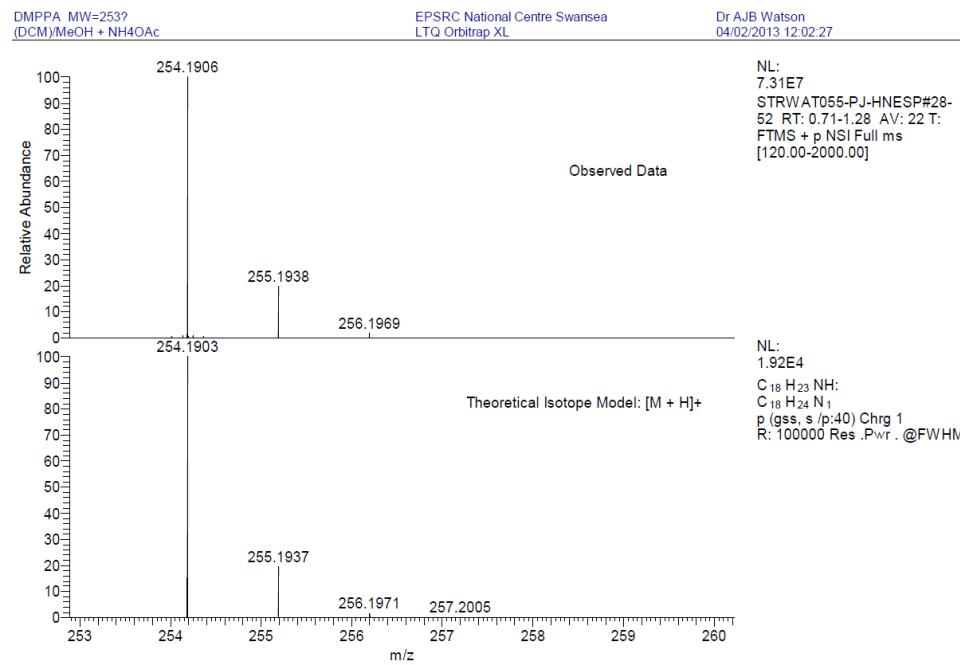
^{13}C NMR:



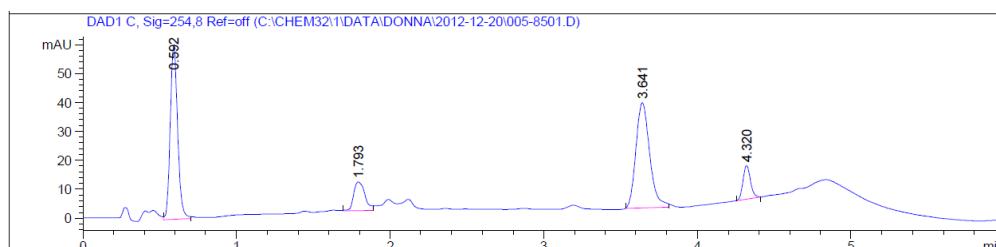
FTIR:



HRMS:



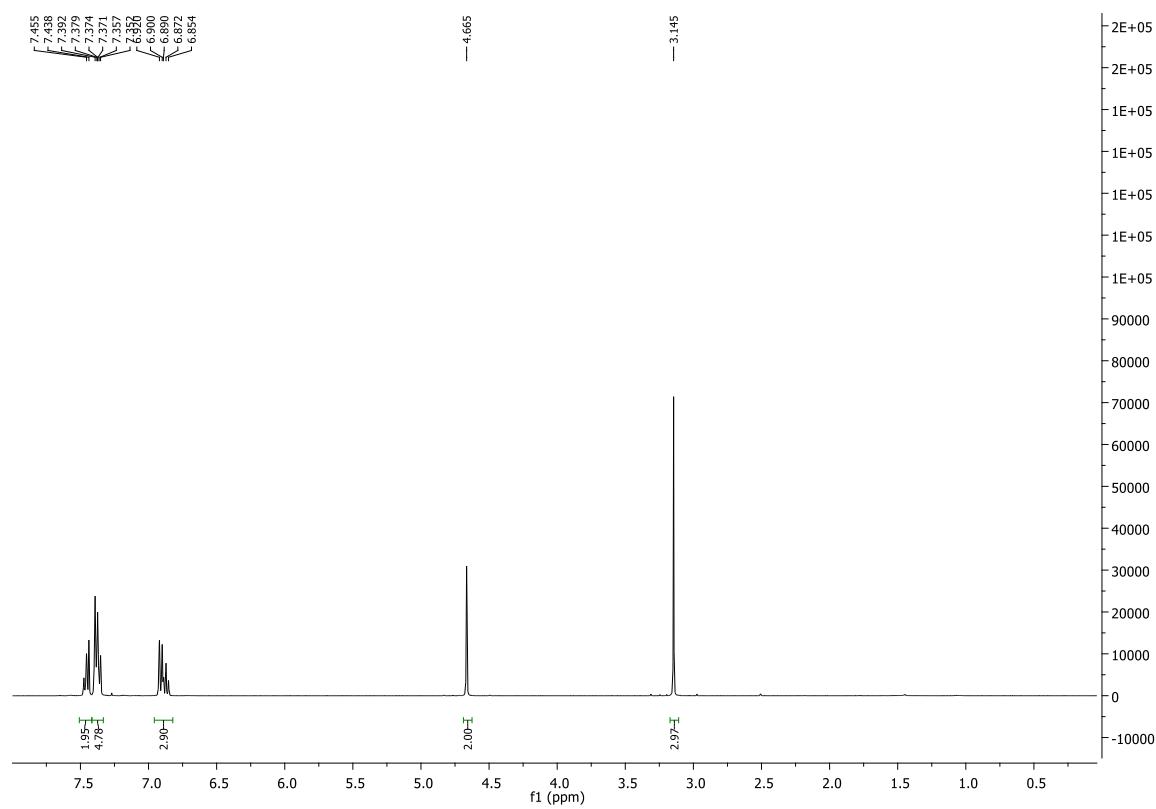
HPLC assay:



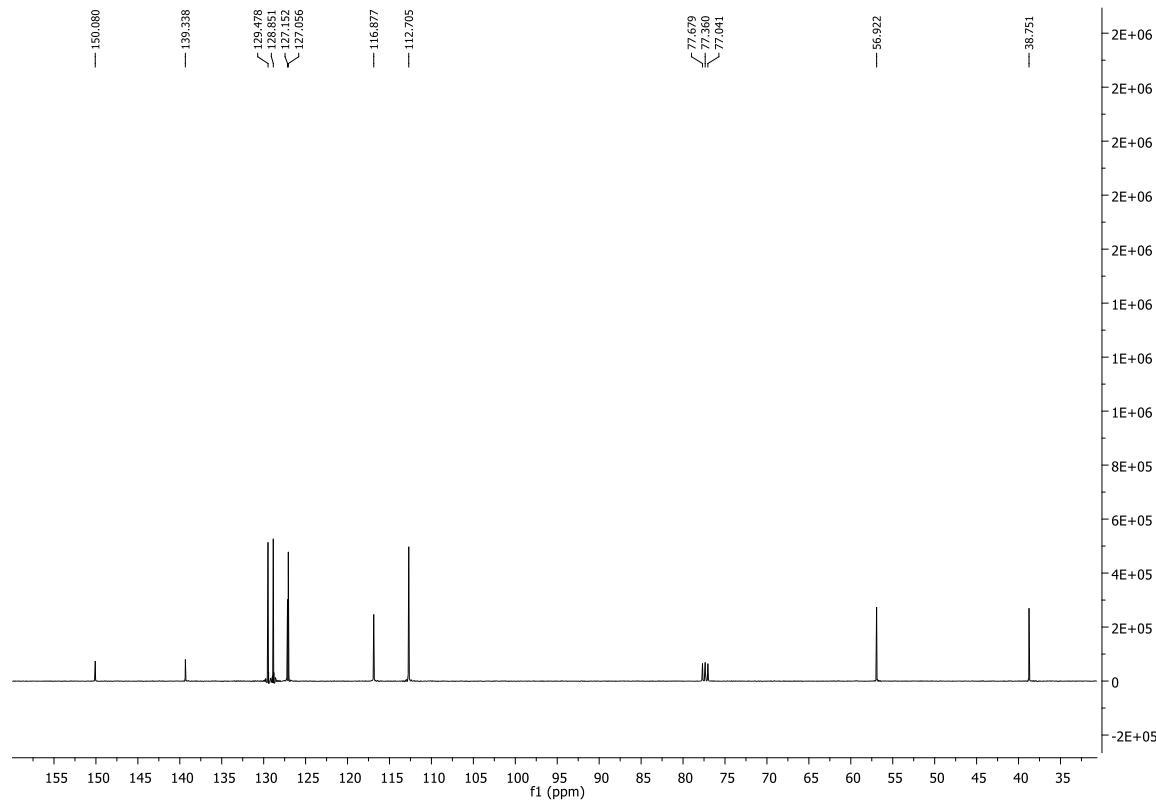
Substrate	R _t (min)
Compound 4: Bis(3-phenylpropyl)amine	4.30
Iodobenzene (standard)	3.64

Compound 5: N-Benzyl-N-methylaniline

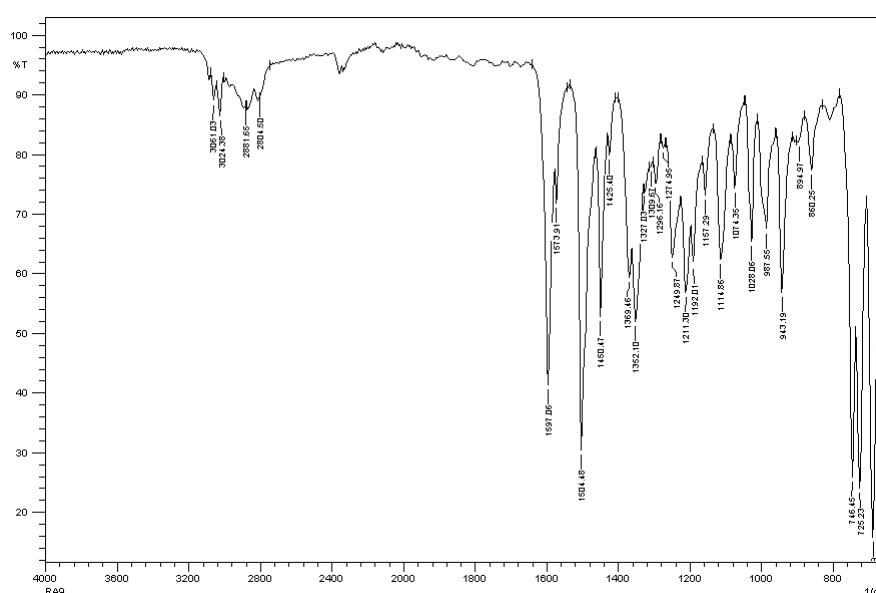
^1H NMR:



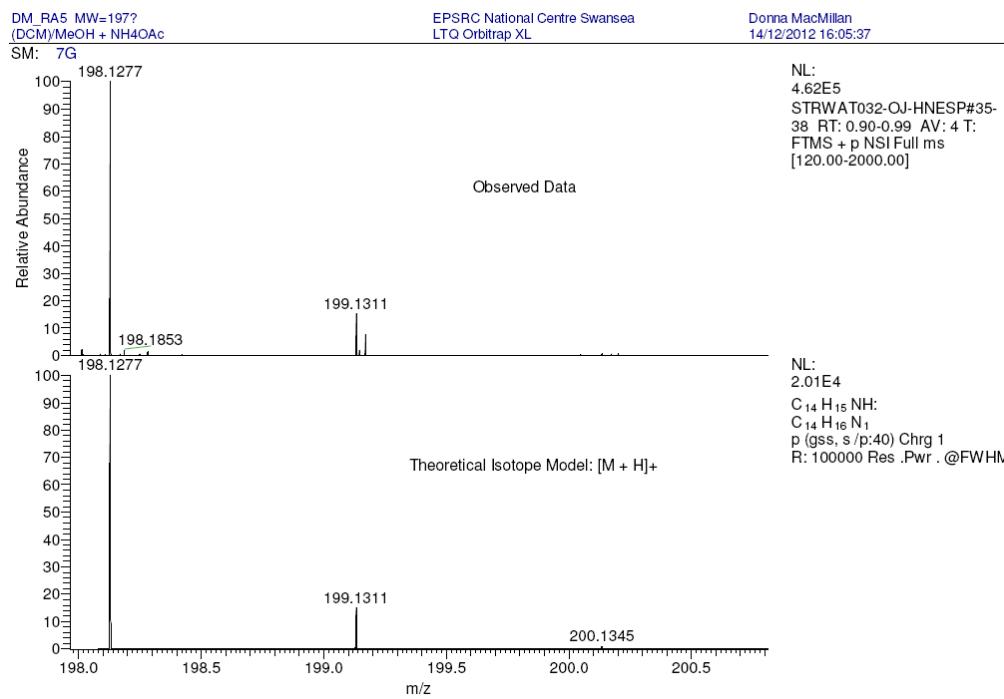
^{13}C NMR:



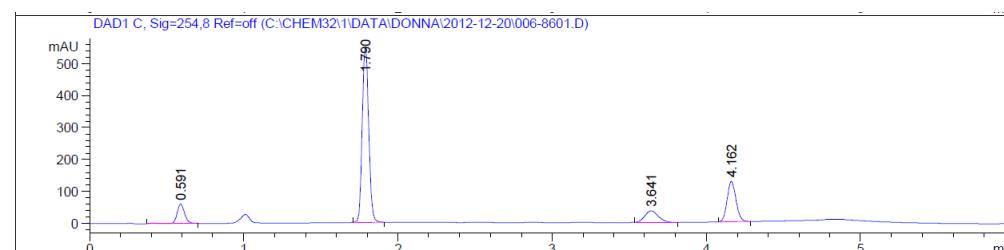
FTIR:



HRMS:



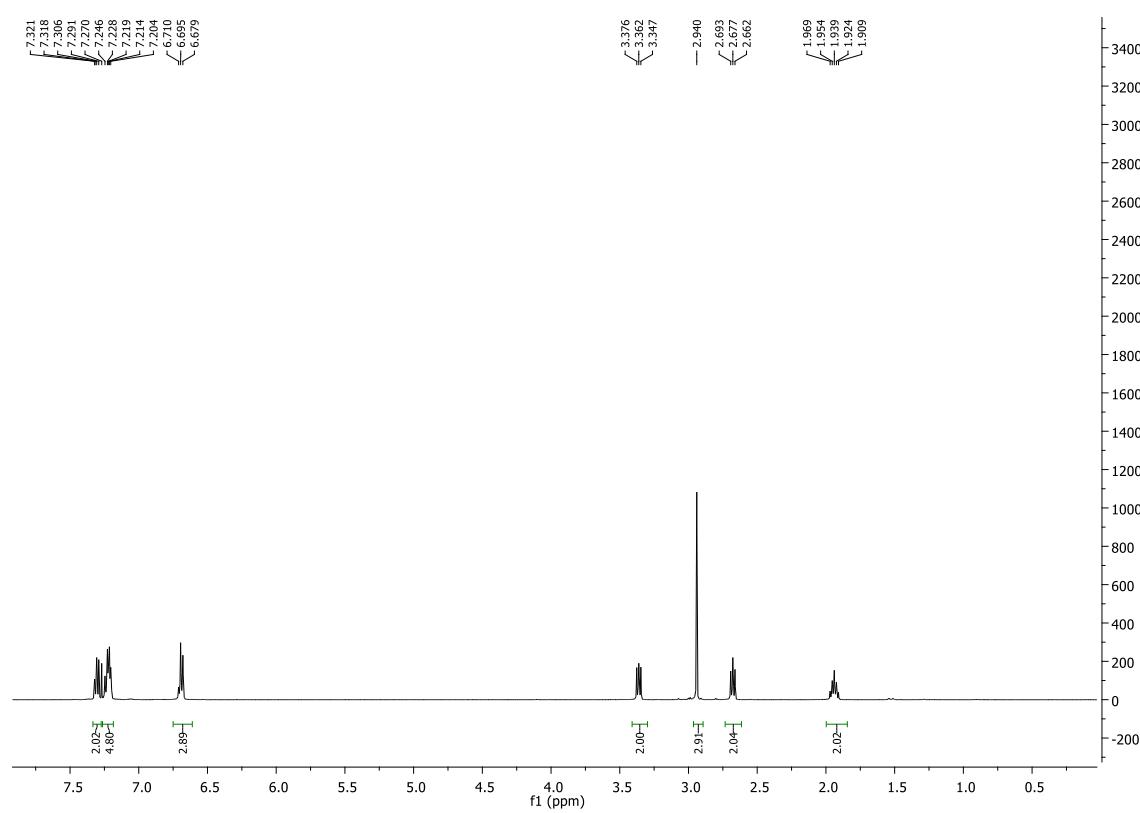
HPLC assay:



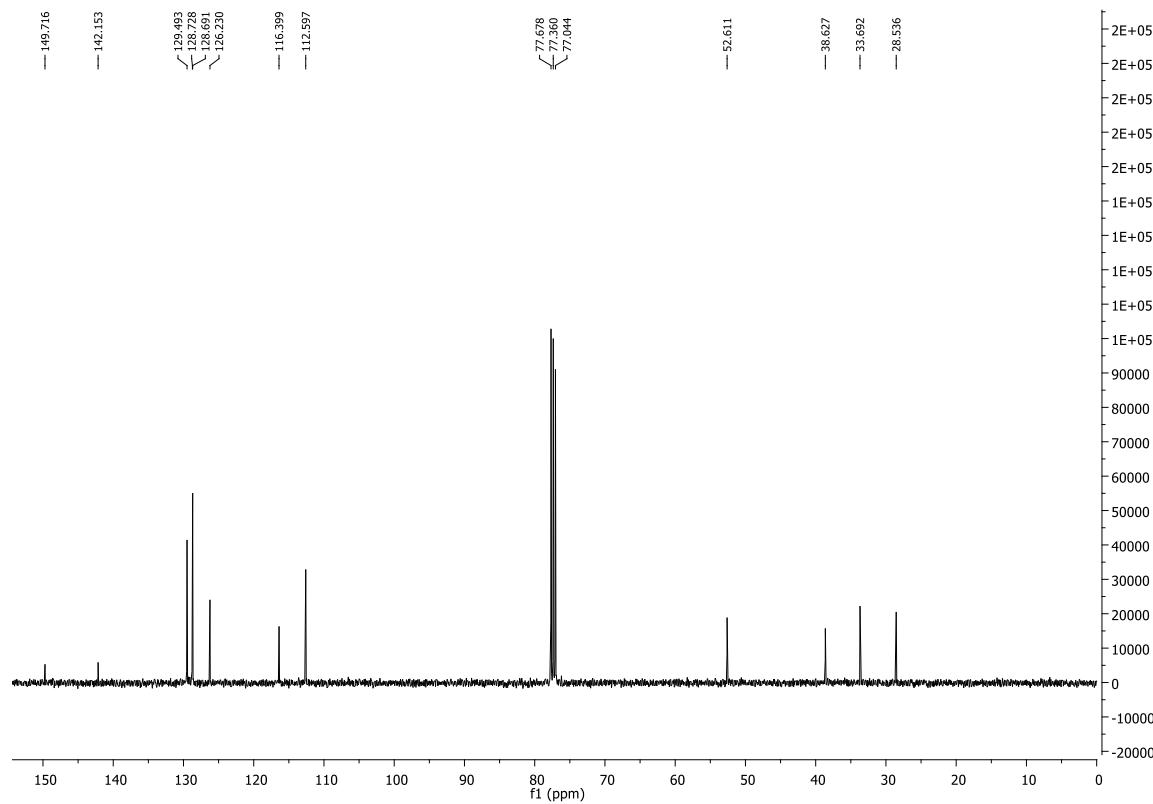
Substrate	R _t (min)
Compound 5: N-benzyl-N-methylaniline	4.16
Iodobenzene (standard)	3.64

Compound 6: N-Methyl-N-(3-phenylpropyl)aniline

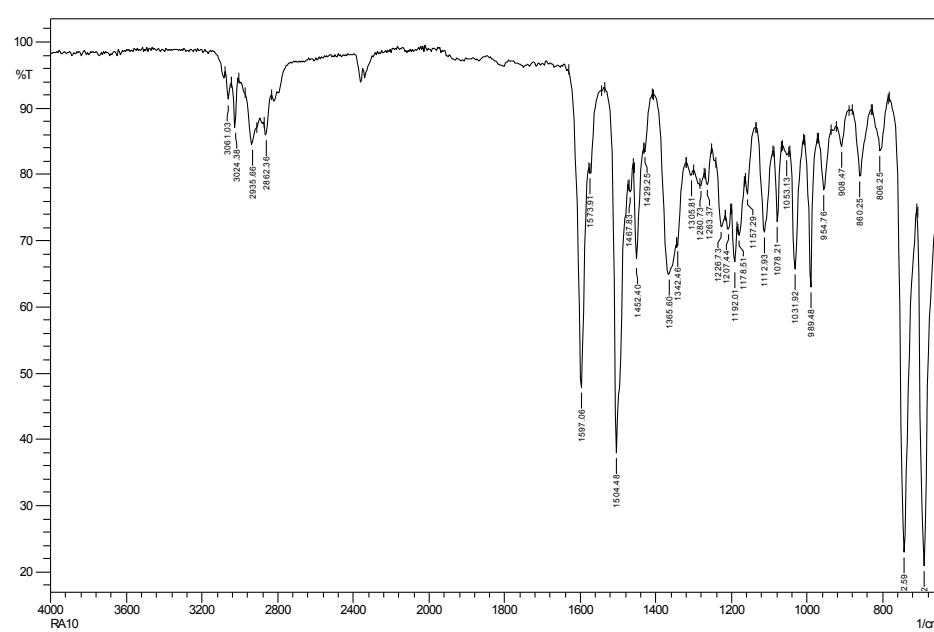
^1H NMR:



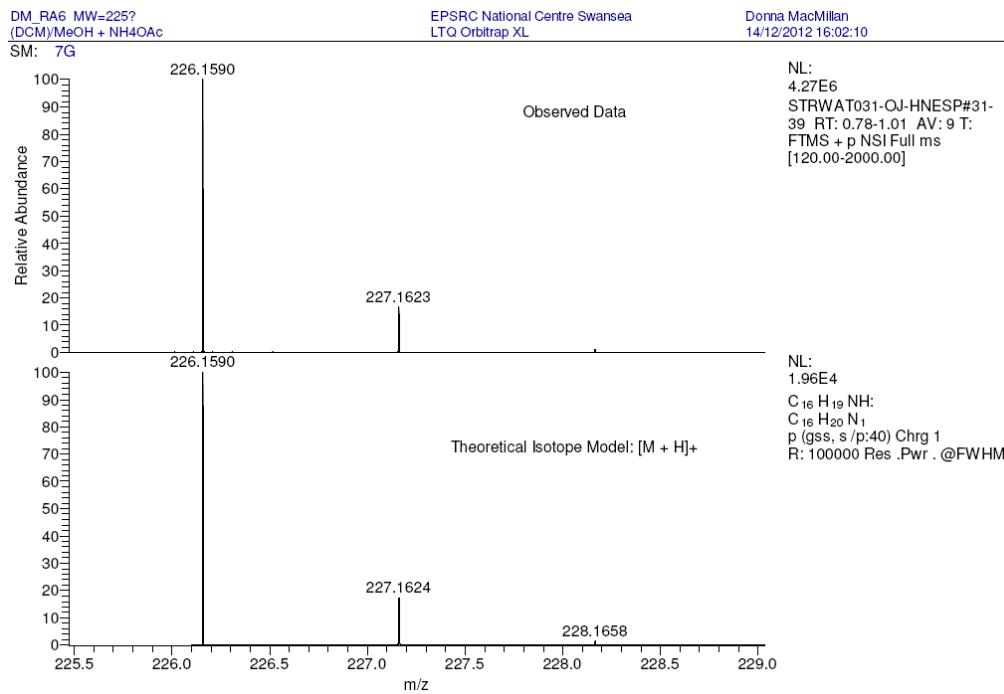
^{13}C NMR:



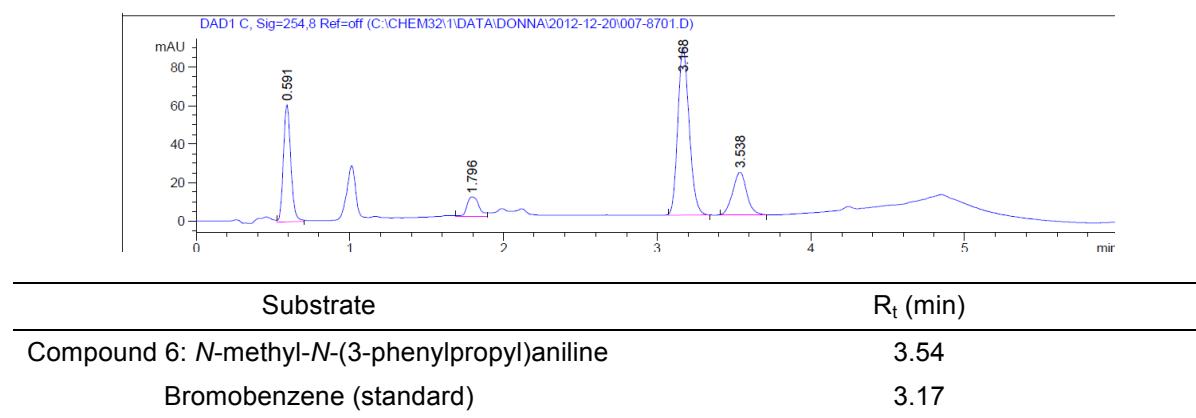
FTIR:



HRMS:

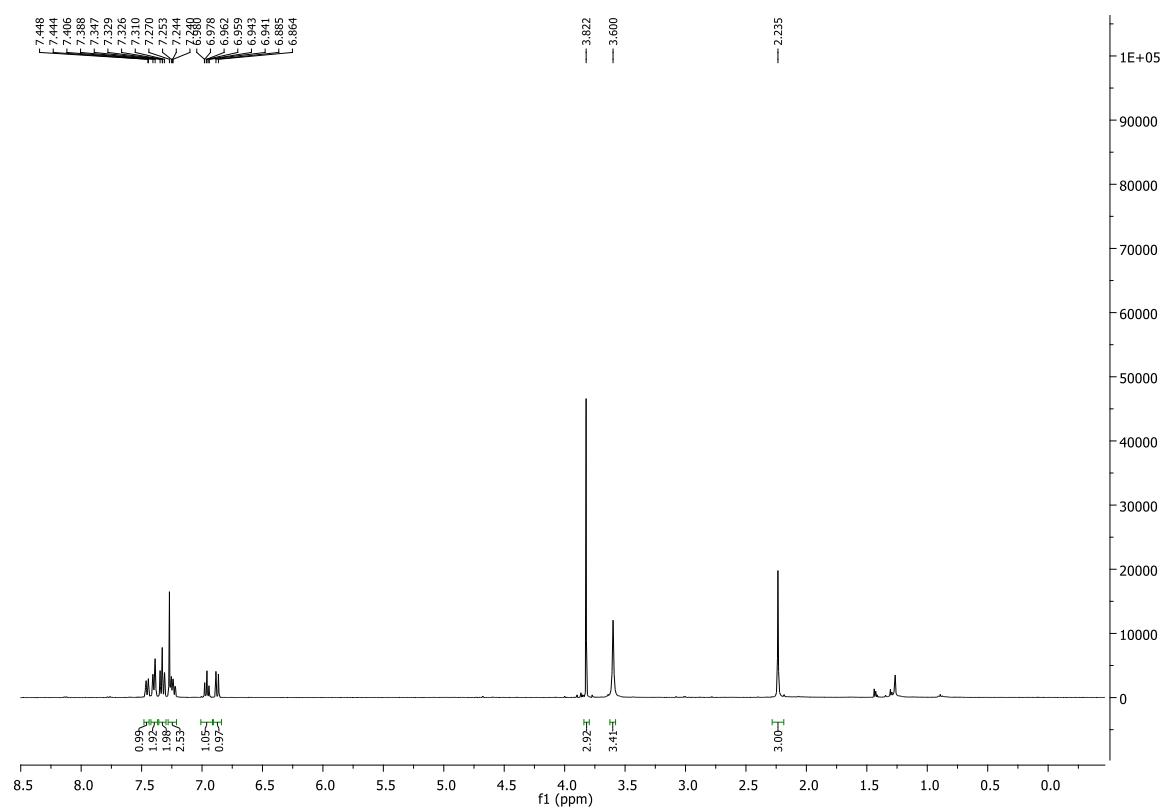


HPLC assay:

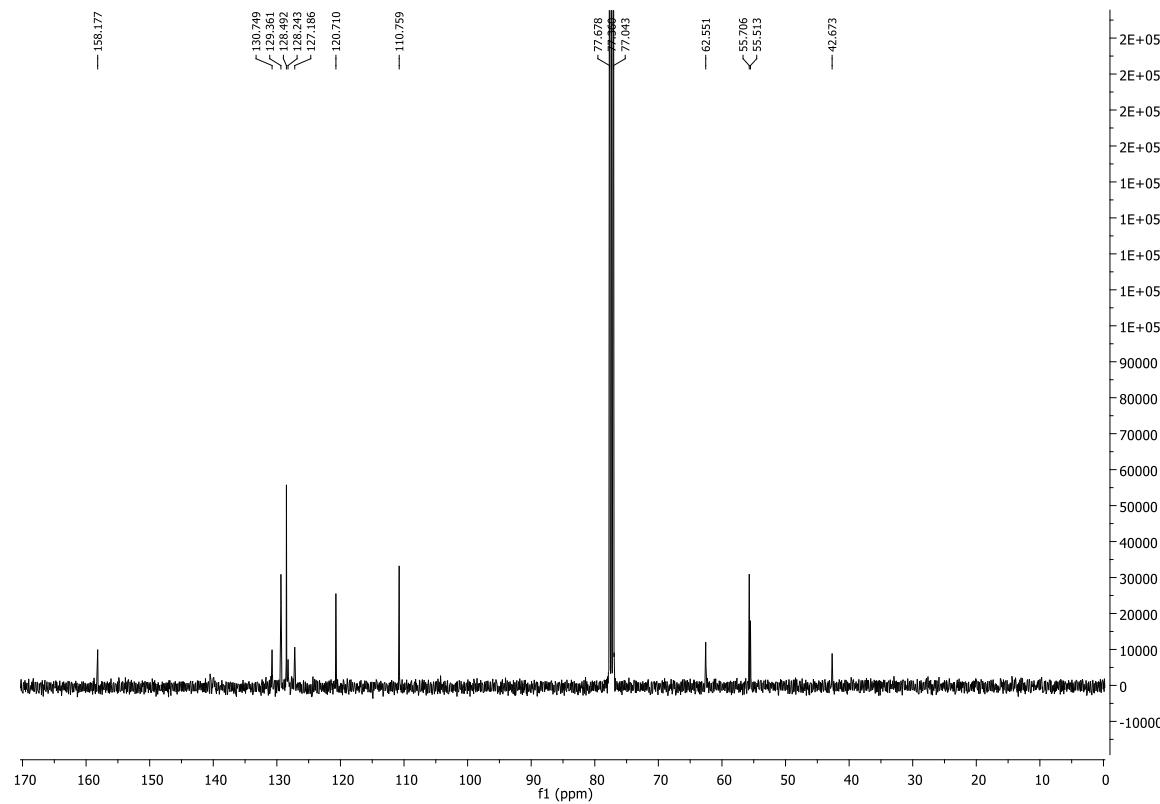


Compound 7: N-Benzyl-1-(2-methoxyphenyl)-N-methylmethanamine

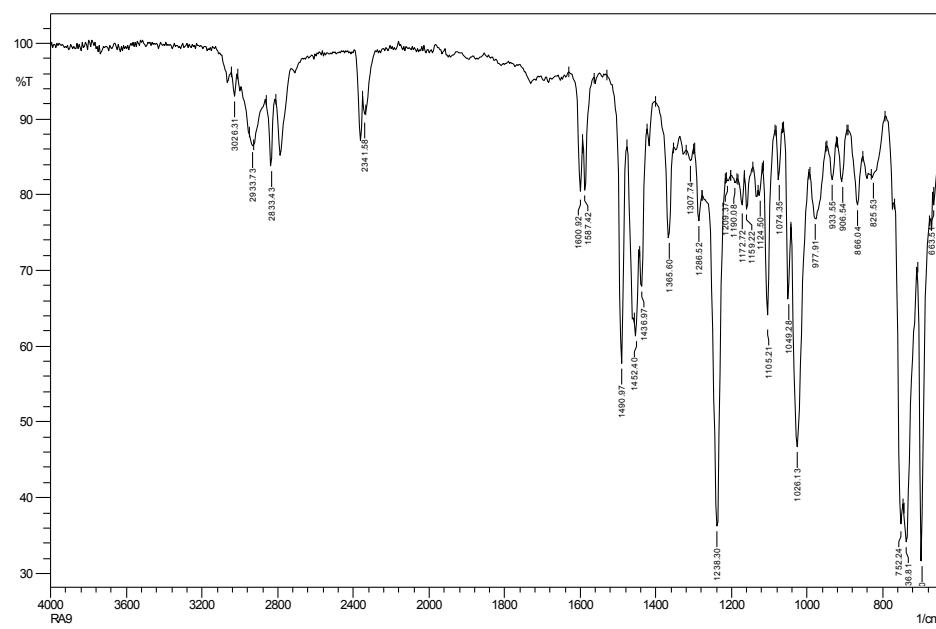
^1H NMR:



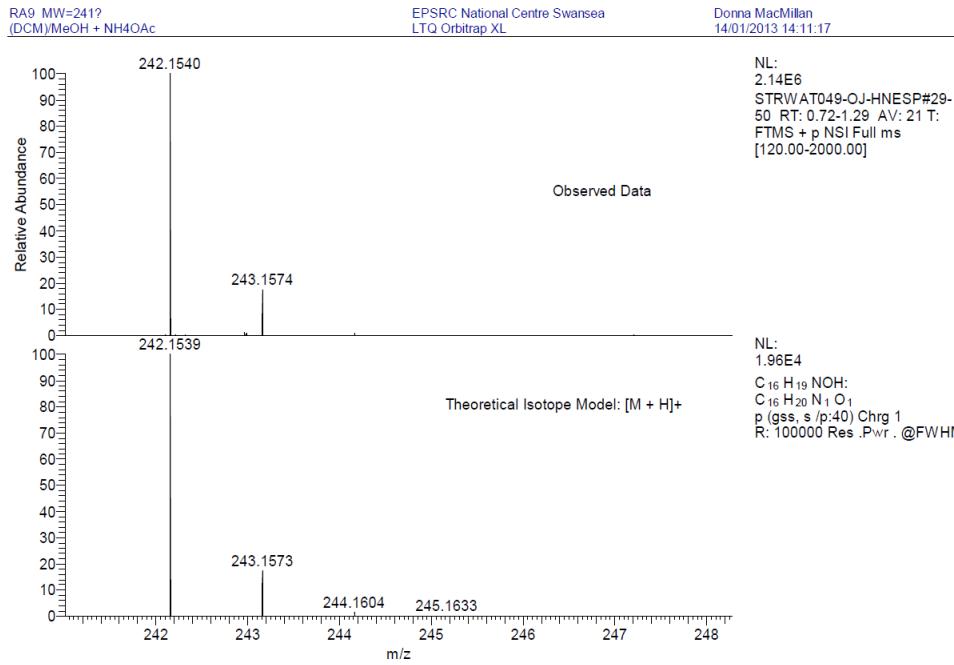
^{13}C NMR:



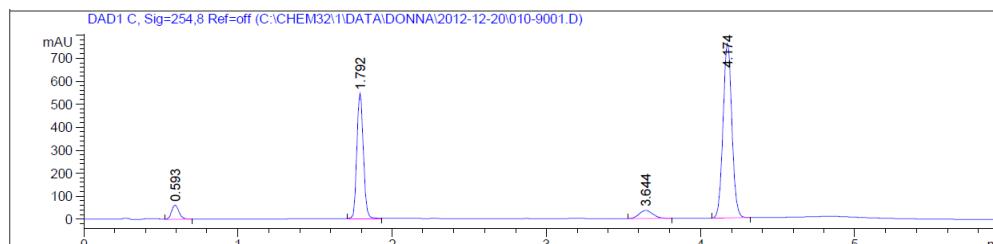
FTIR:



HRMS:



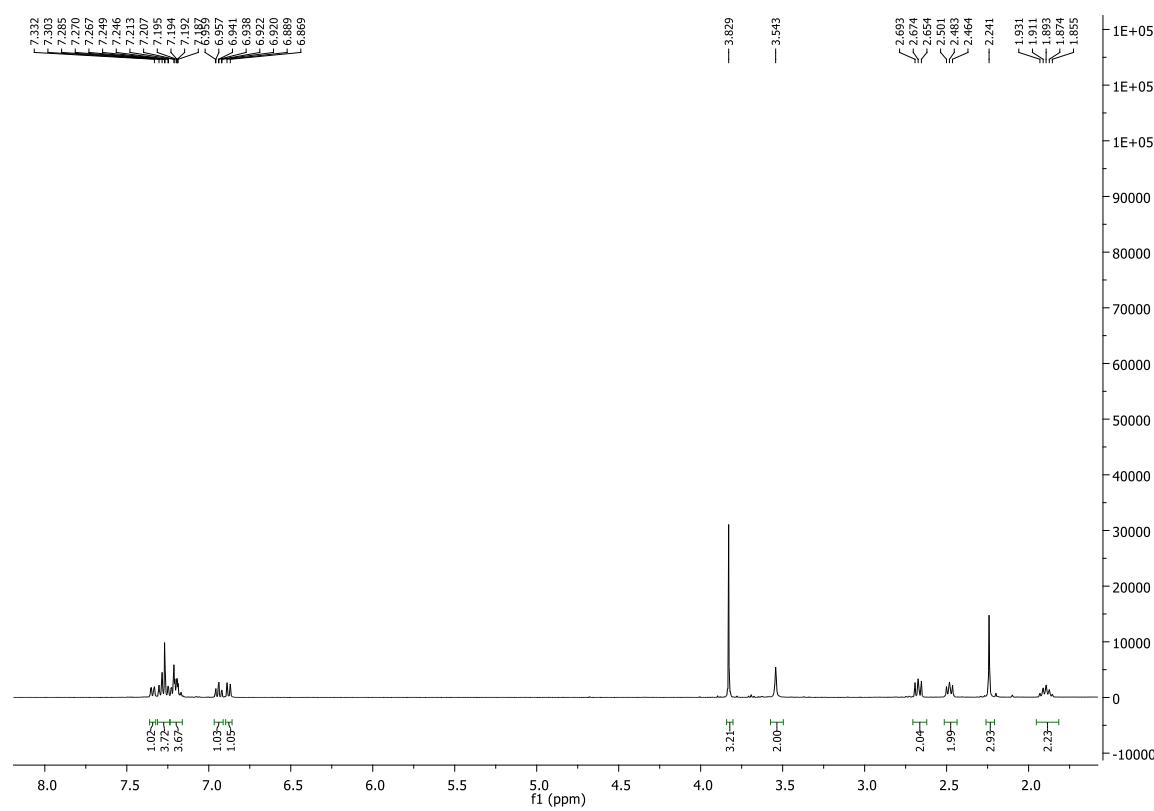
HPLC assay:



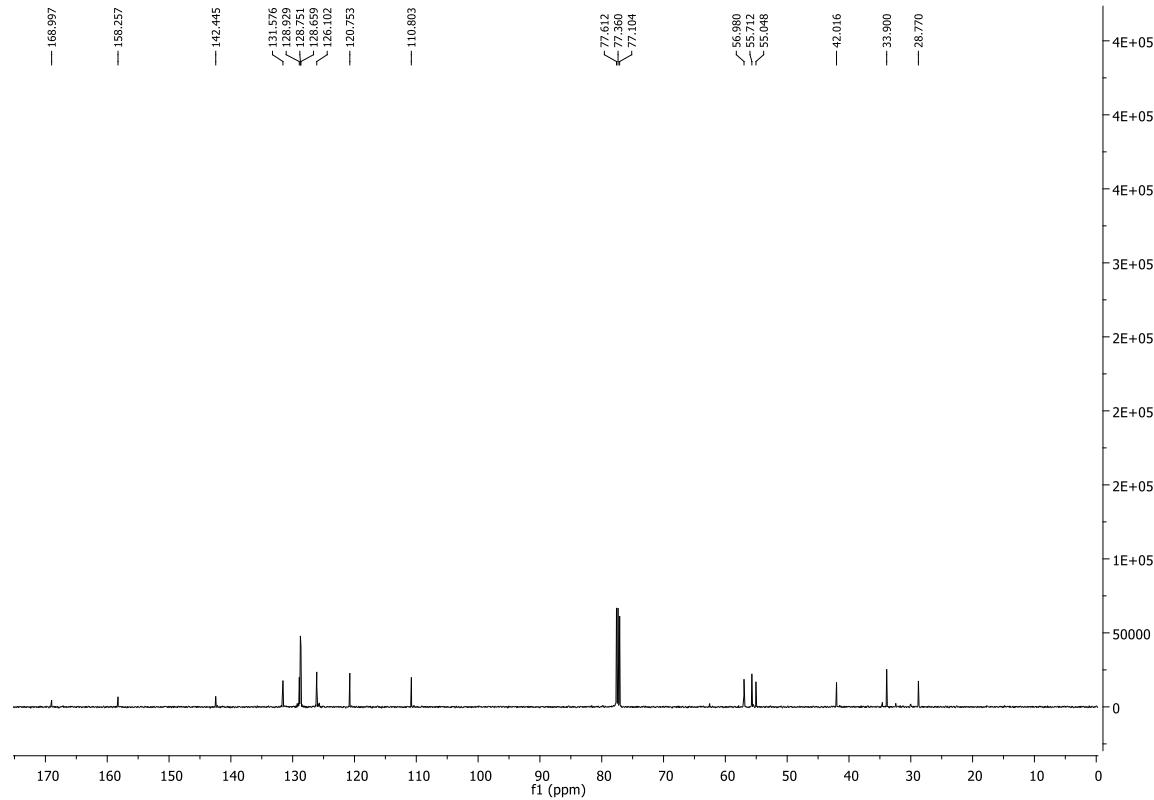
Substrate	R _t (min)
Compound 9: N-benzyl-1-(2-methoxyphenyl)-N-methylmethanamine	4.17
Iodobenzene (standard)	3.64

Compound 8: N-(2-Methoxybenzyl)-N-methyl-3-phenylpropan-1-amine

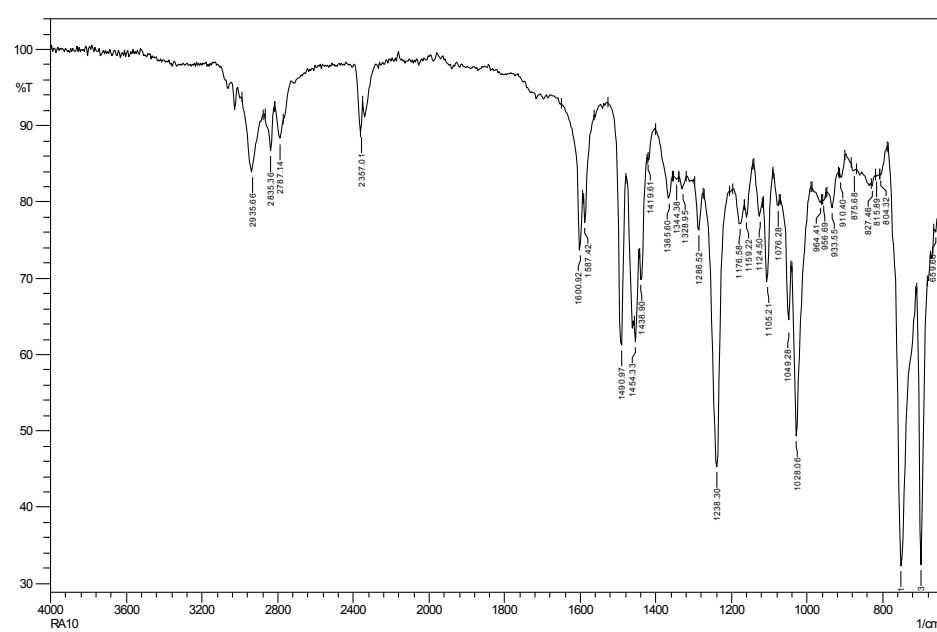
^1H NMR:



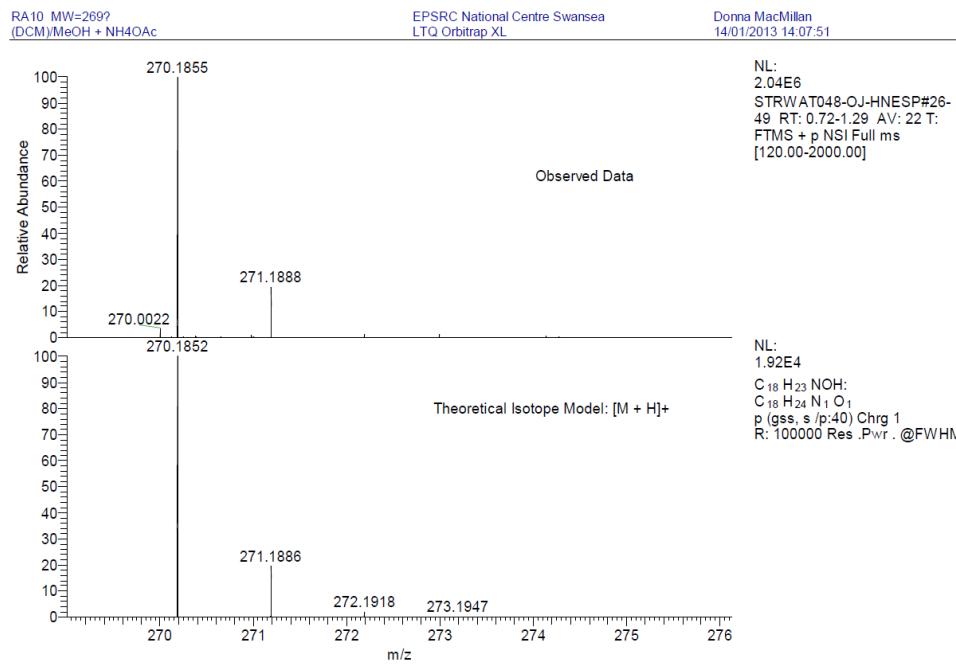
^{13}C NMR:



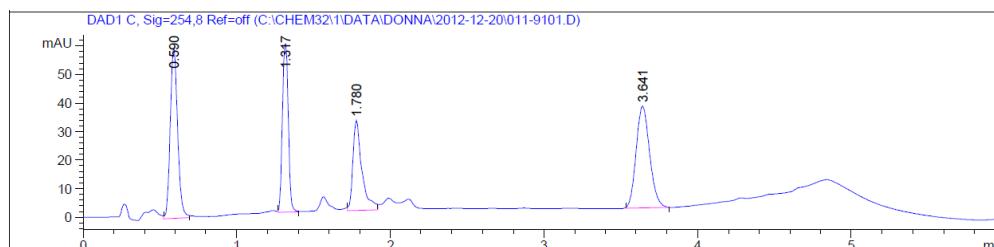
FTIR:



HRMS:



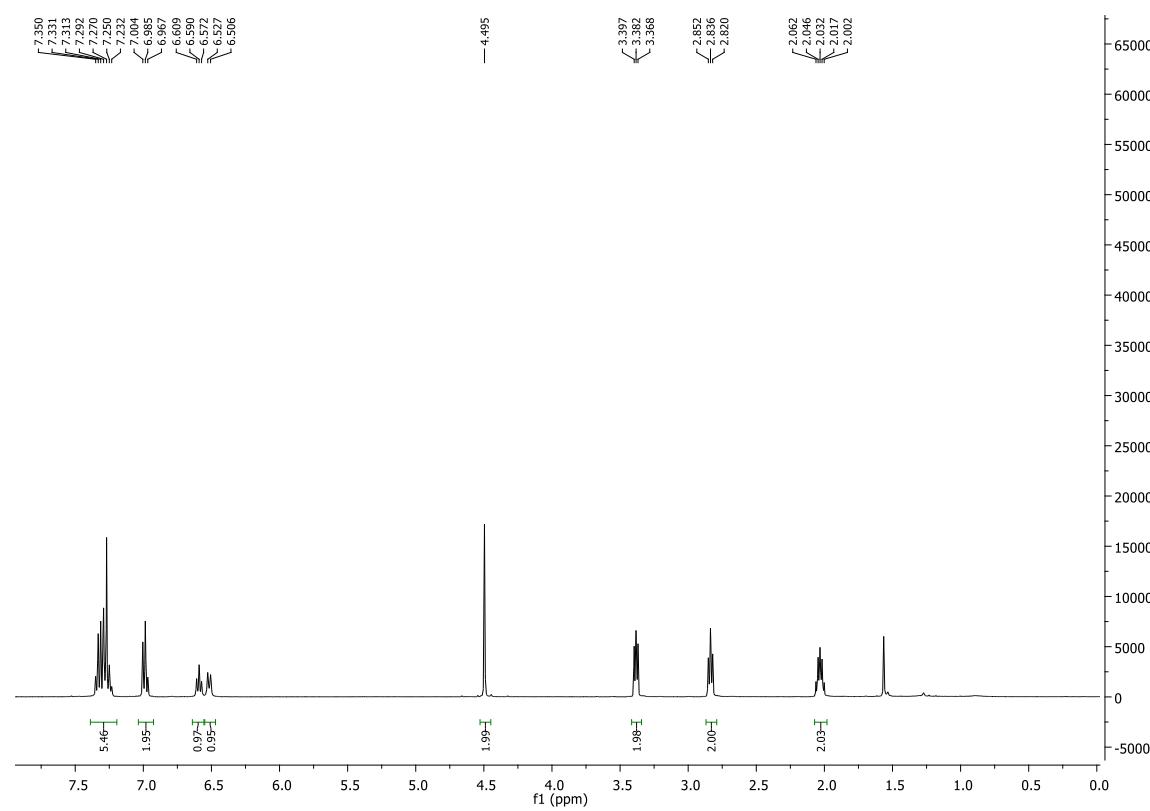
HPLC assay:



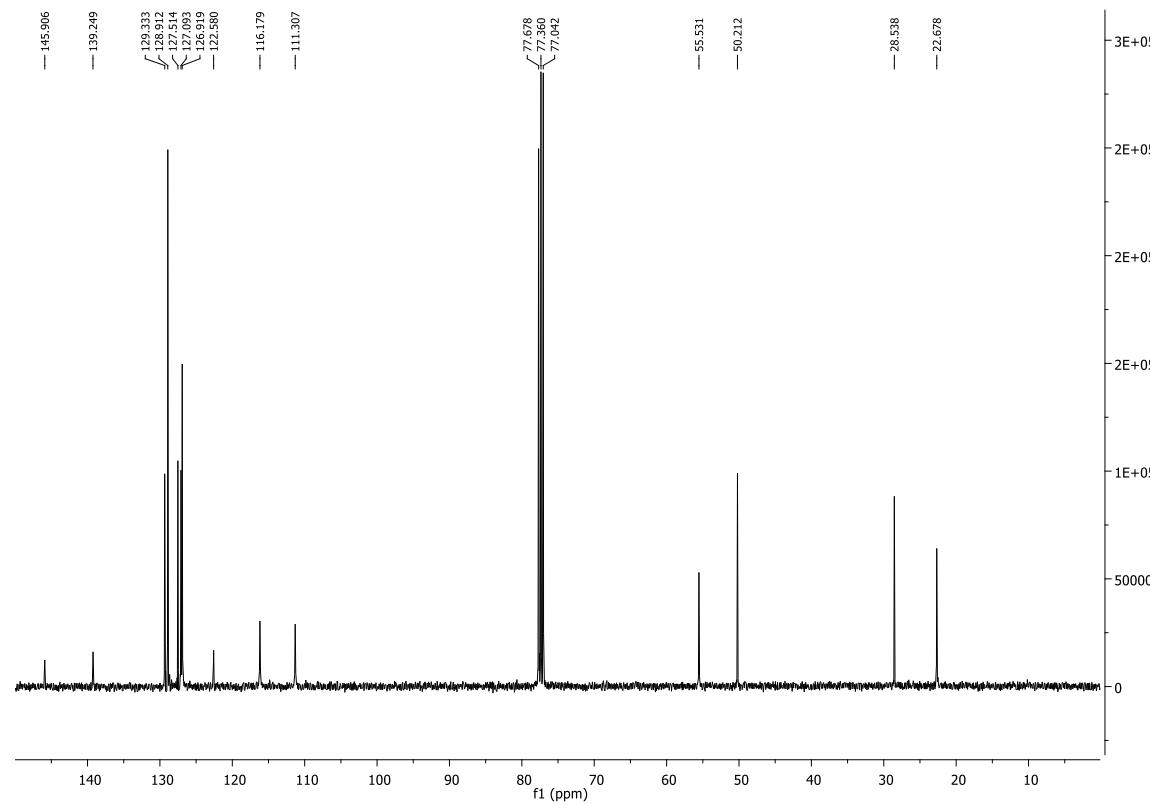
Substrate	R _t (min)
Compound 10: N-(2-methoxybenzyl)-N-methyl-3-phenylpropan-1-amine	1.32
Iodobenzene (standard)	3.64

Compound 9: 1-Benzyl-1,2,3,4-tetrahydroquinoline

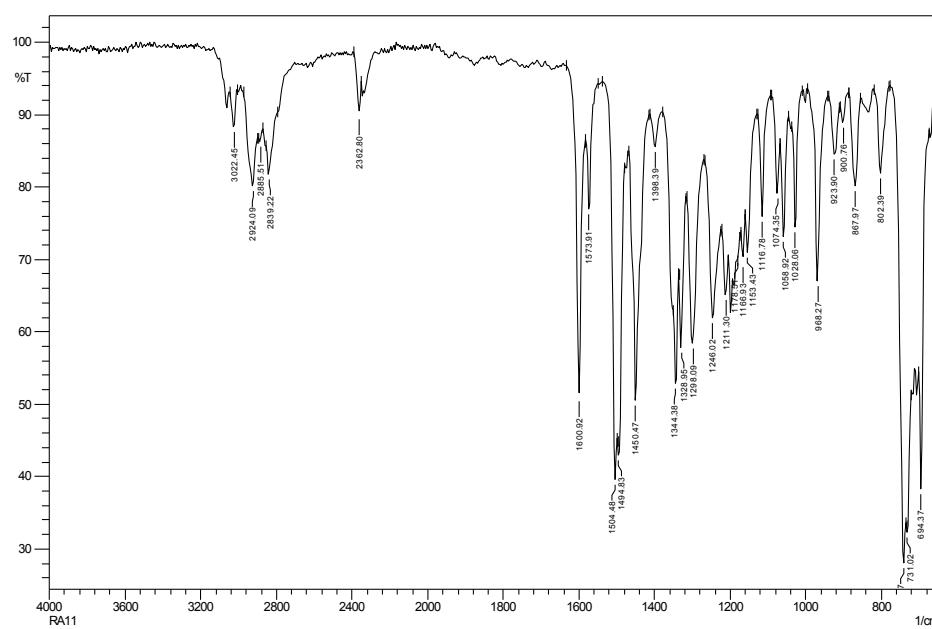
^1H NMR:



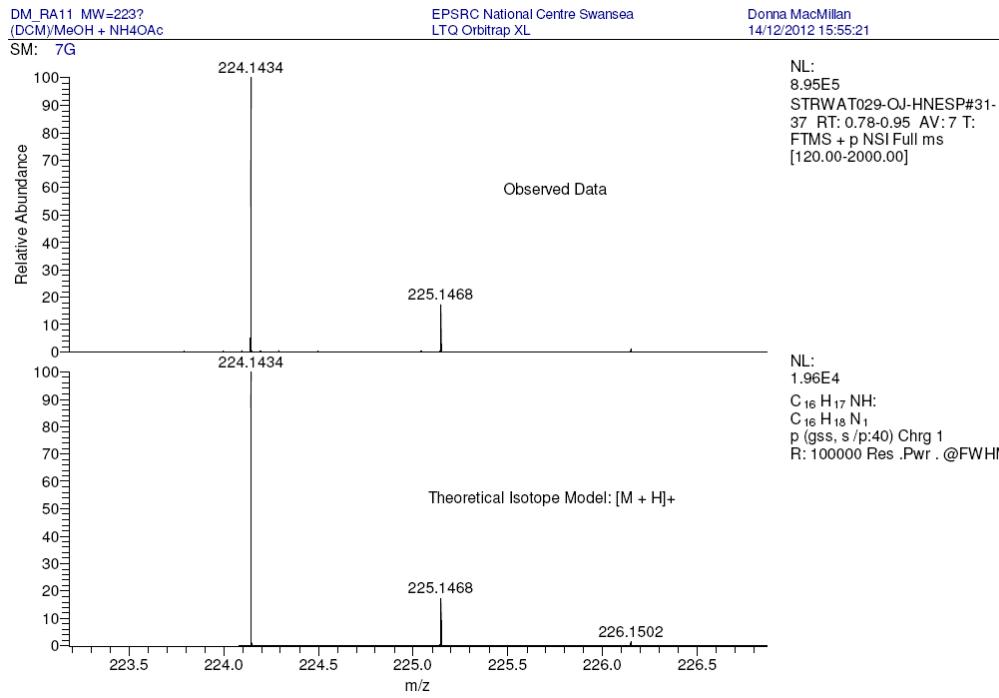
^{13}C NMR:



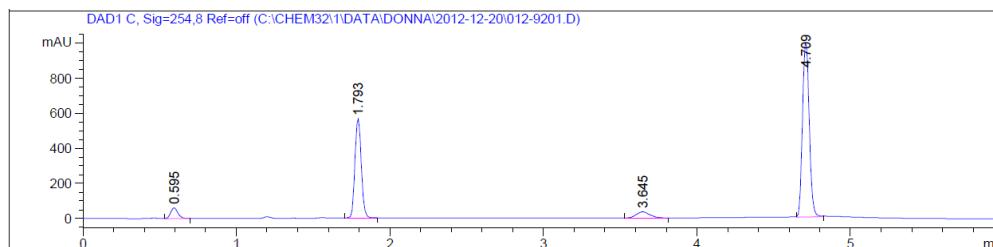
FTIR:



HRMS:



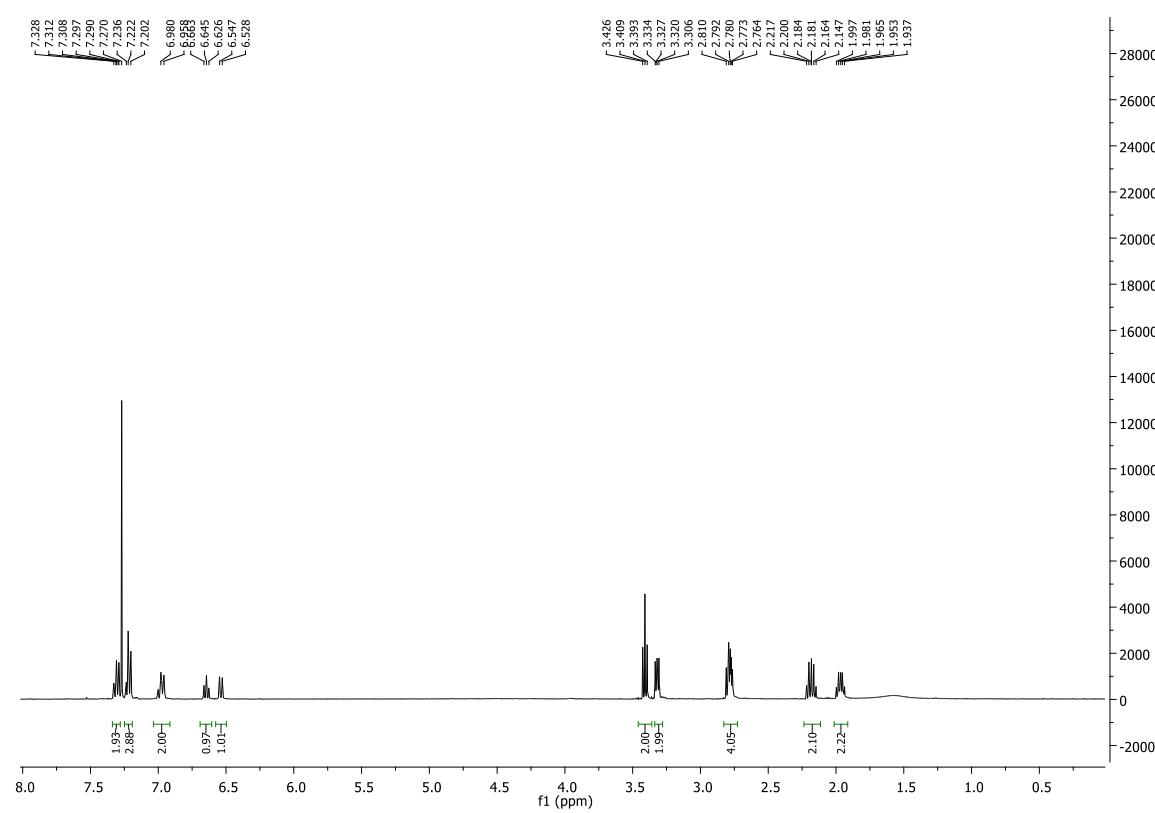
HPLC assay:



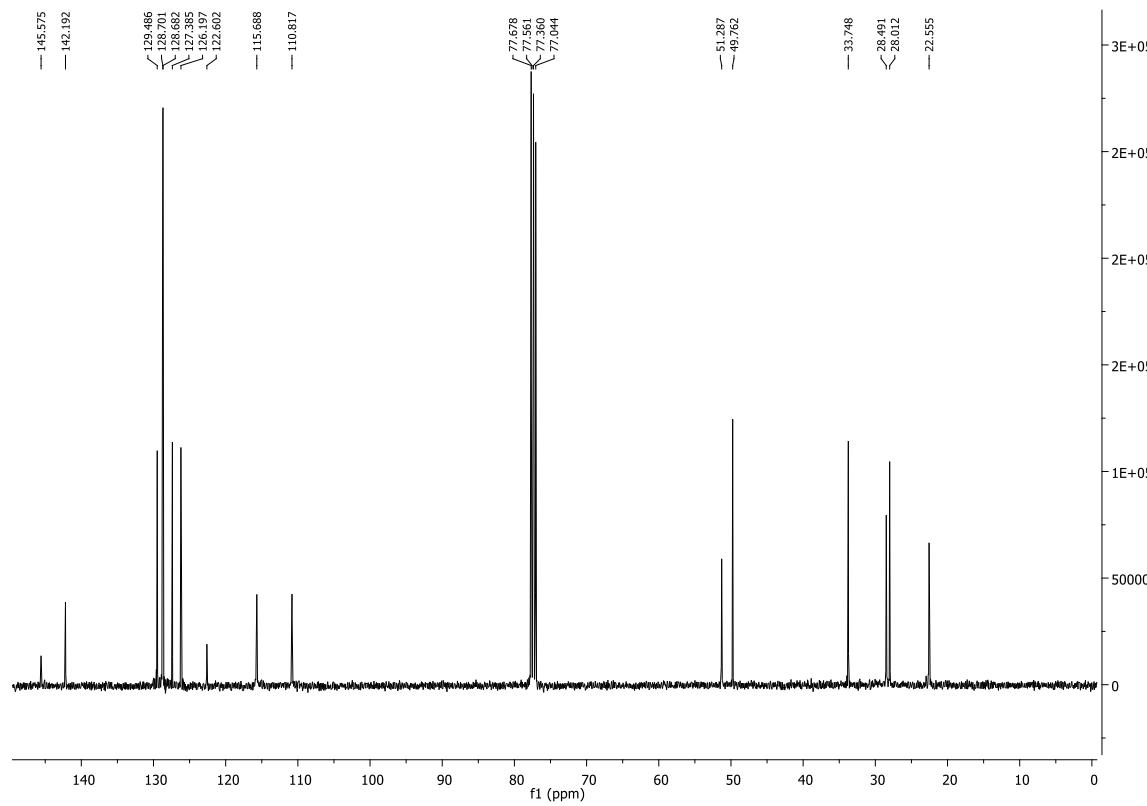
Substrate	R _t (min)
Compound 11: 1-Benzyl-1,2,3,4-tetrahydroquinoline	4.71
Iodobenzene (standard)	3.64

Compound 10: 1-(3-Phenylpropyl)-1,2,3,4-tetrahydroquinoline

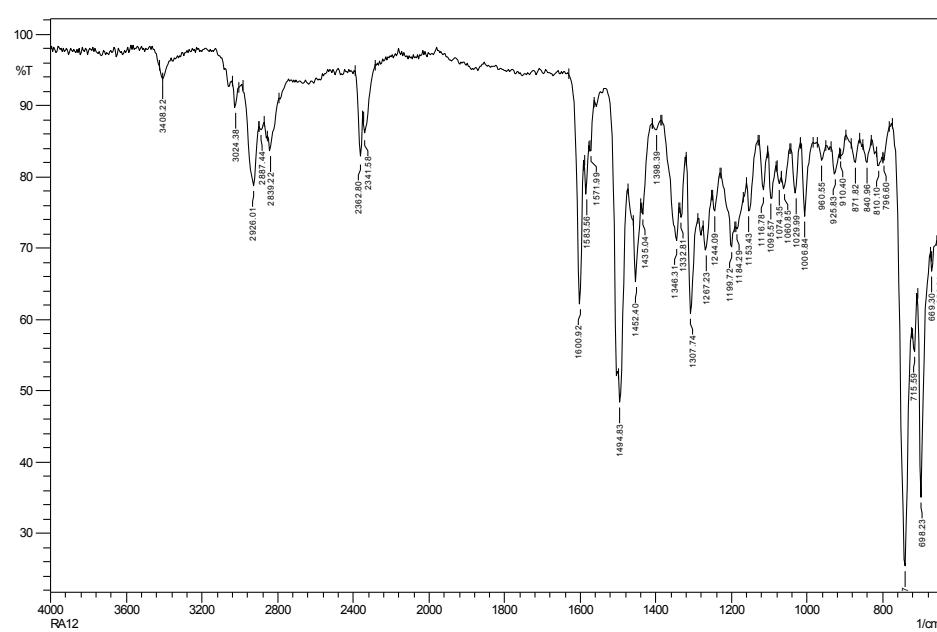
¹H NMR:



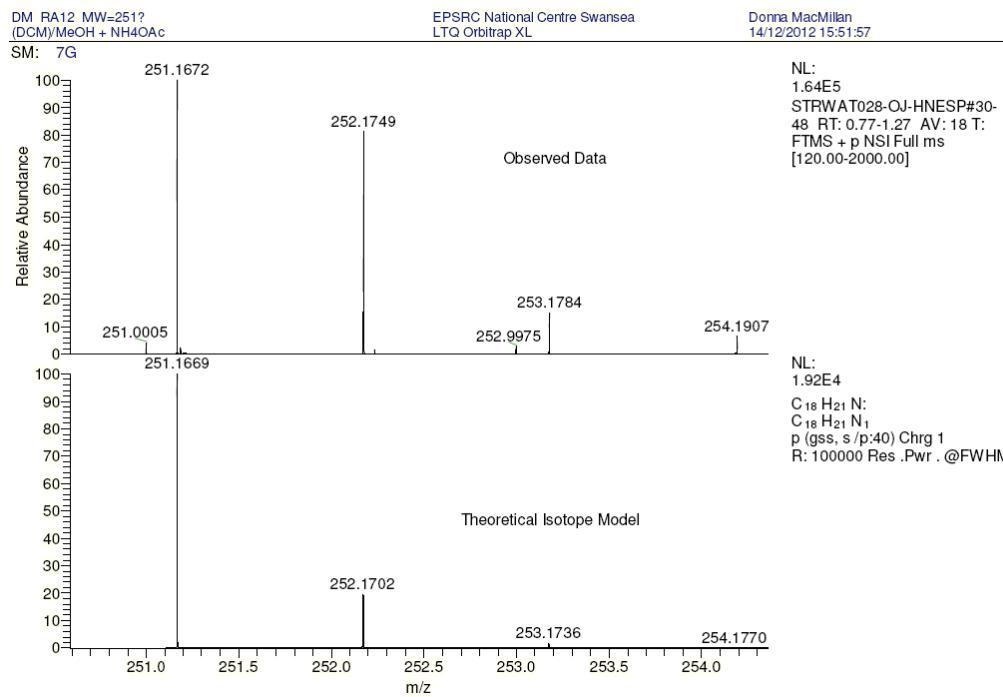
¹³C NMR:



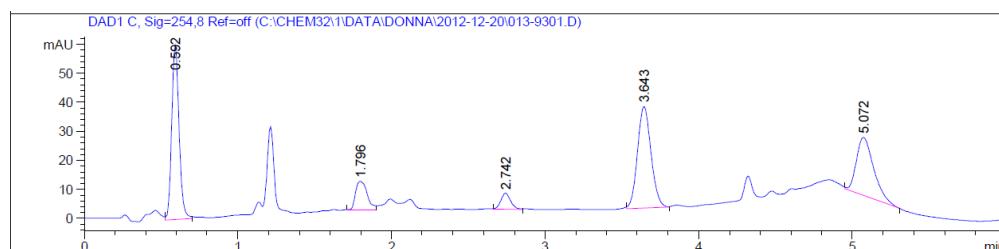
FTIR:



HRMS:



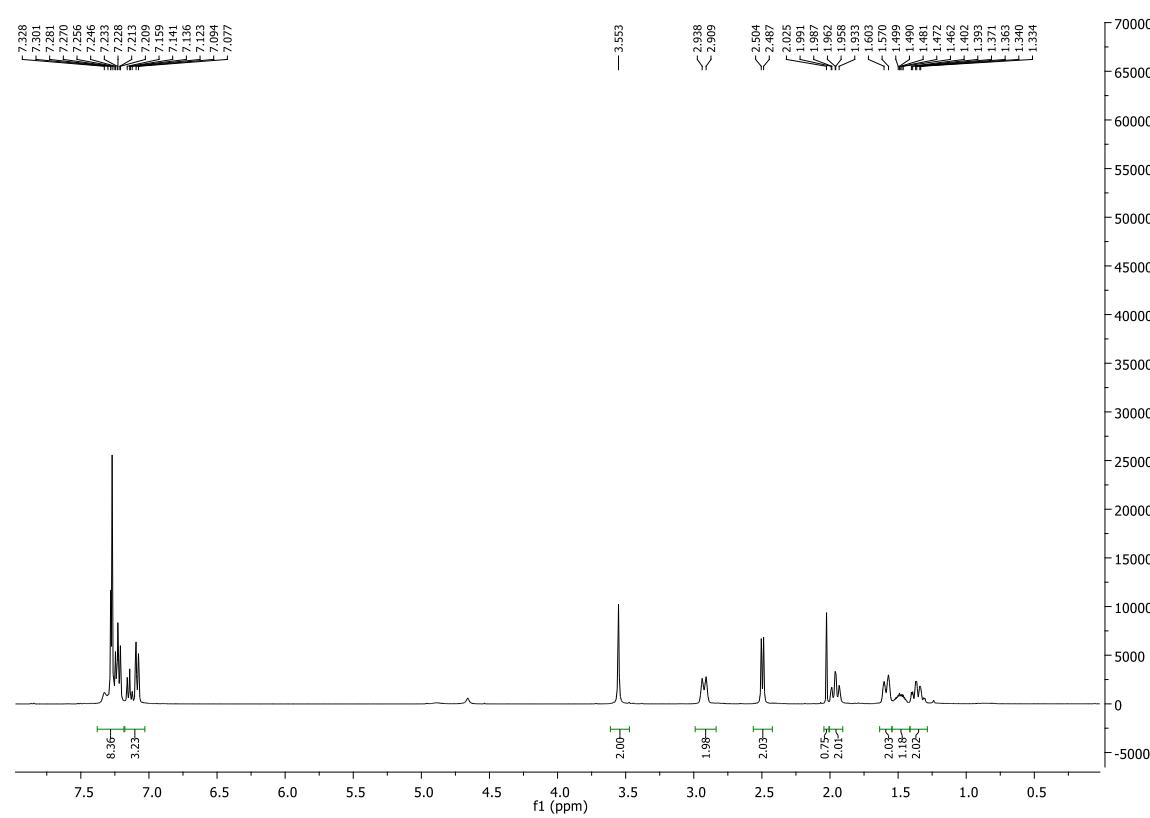
HPLC assay:



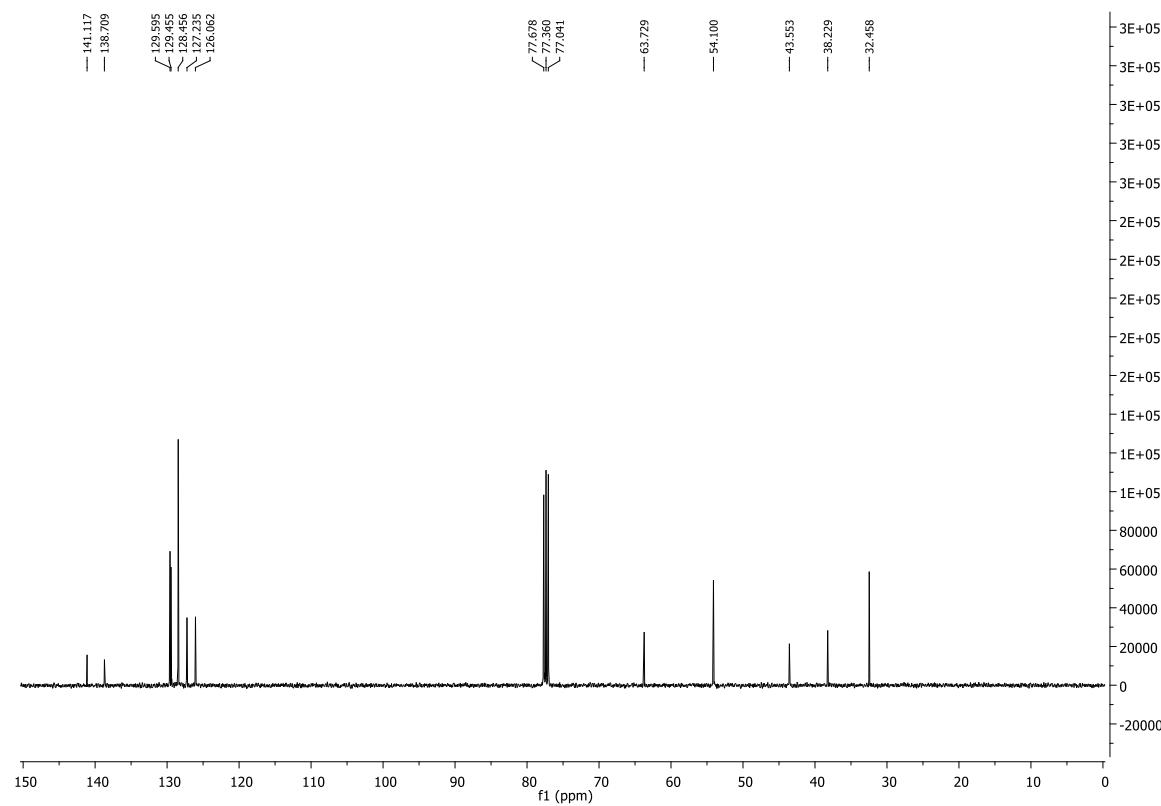
Substrate	R _t (min)
Compound 12: 1-(3-Phenylpropyl)-1,2,3,4-tetrahydroquinoline	2.74
Iodobenzene (standard)	3.64

Compound 11: 1,4-Dibenzylpiperidine

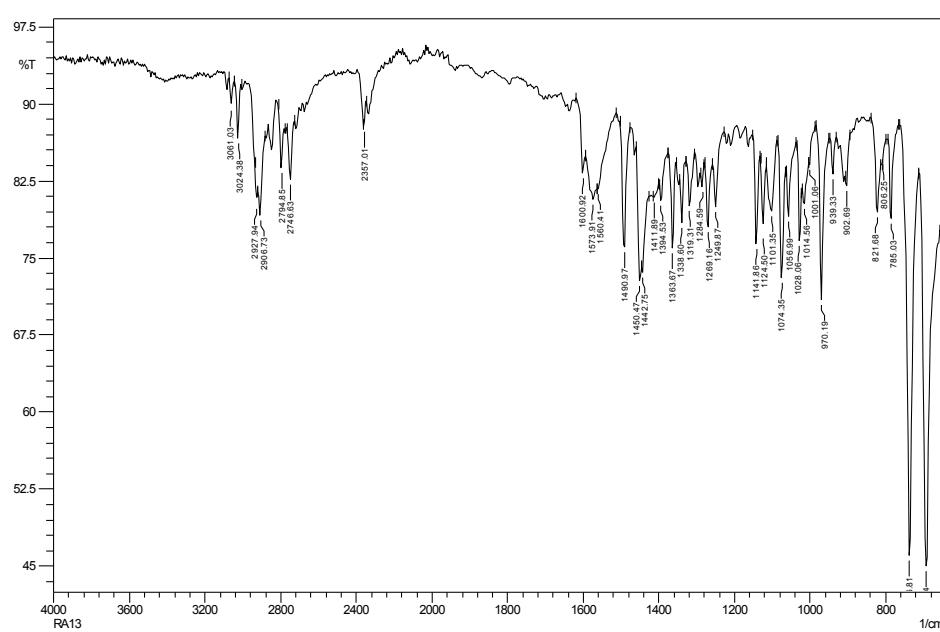
^1H NMR:



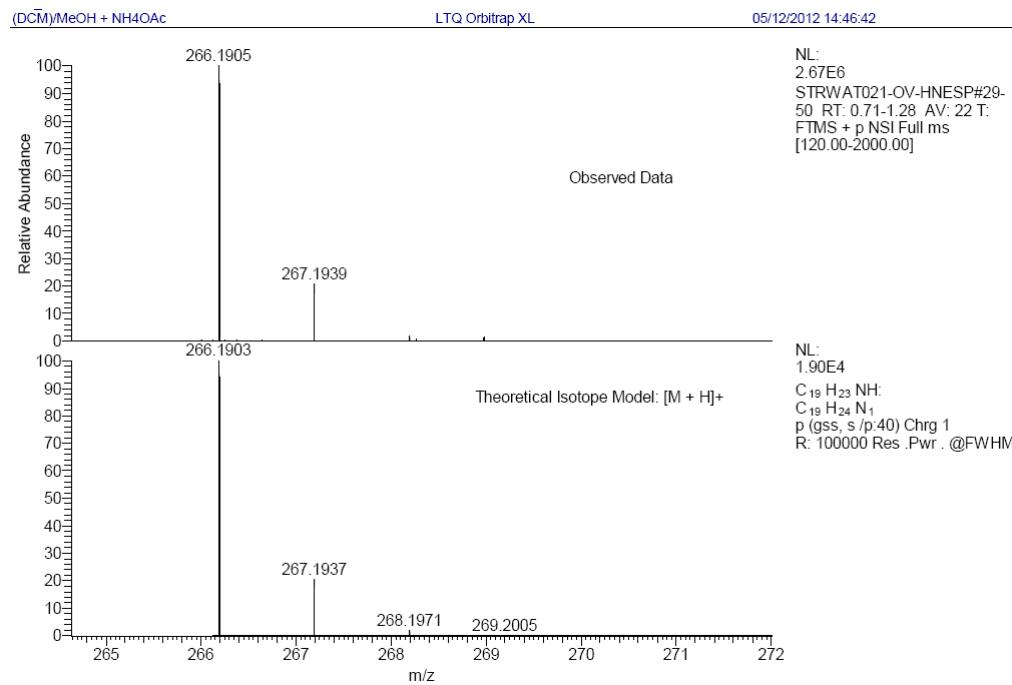
^{13}C NMR:



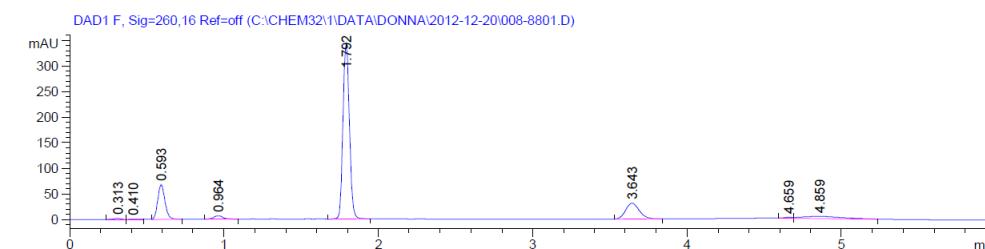
FTIR:



HRMS:



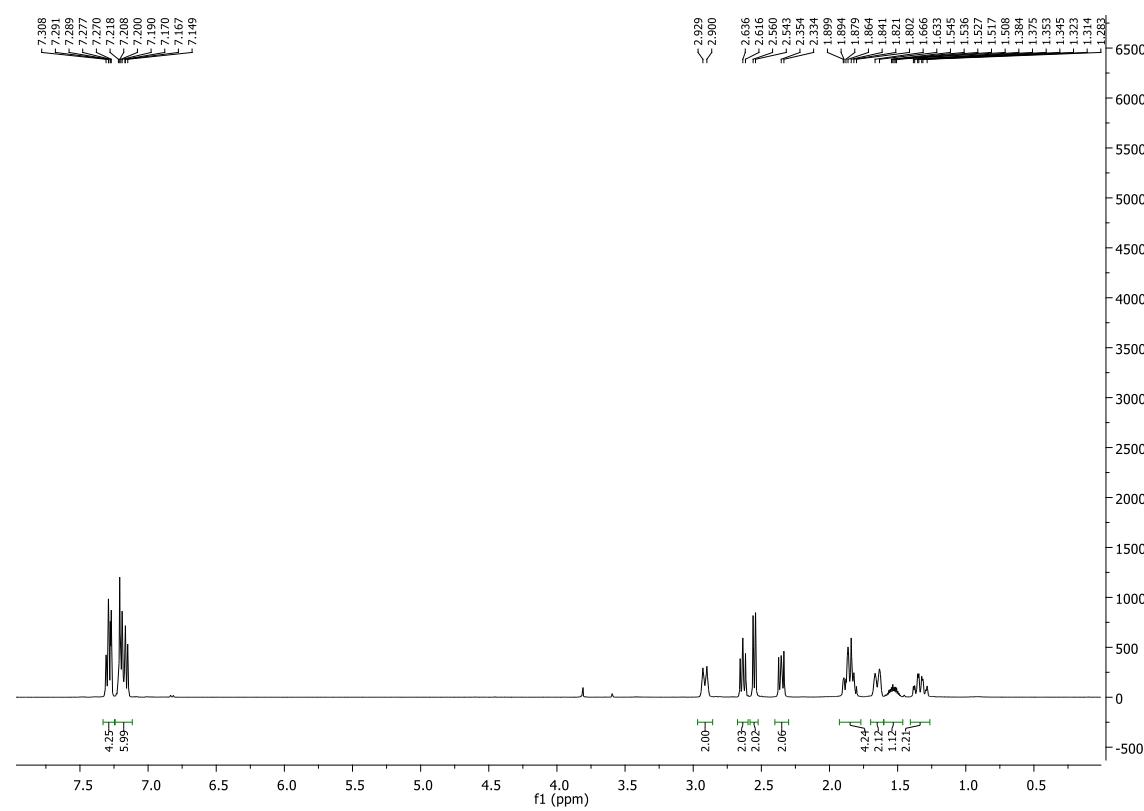
HPLC assay:



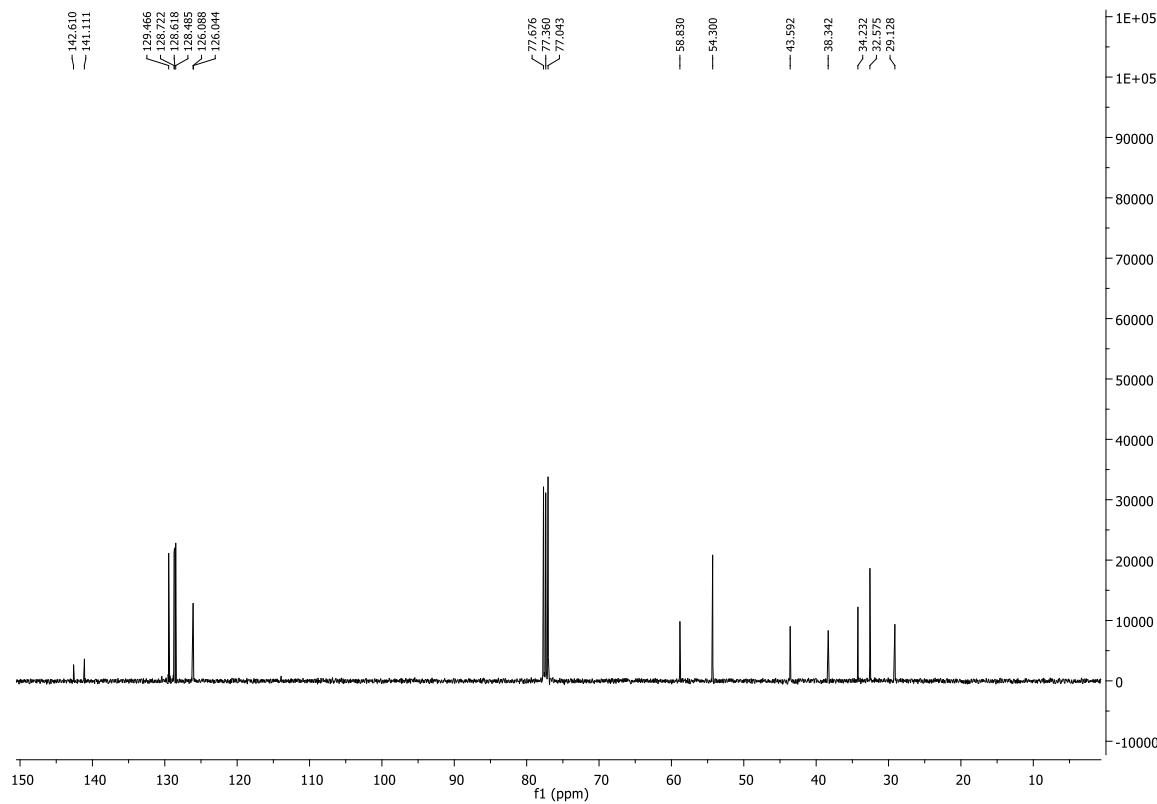
Substrate	R _t (min)
Compound 7: 1,4-Dibenzylpiperidine	0.96
Iodobenzene (standard)	3.64

Compound 12: 4-Benzyl-1-(3-phenylpropyl)piperidine

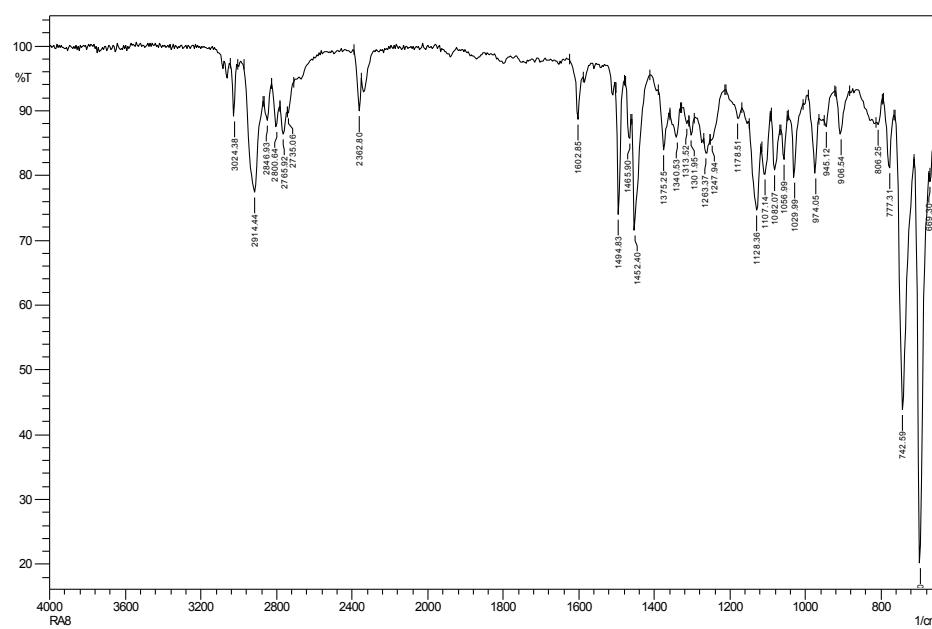
^1H NMR:



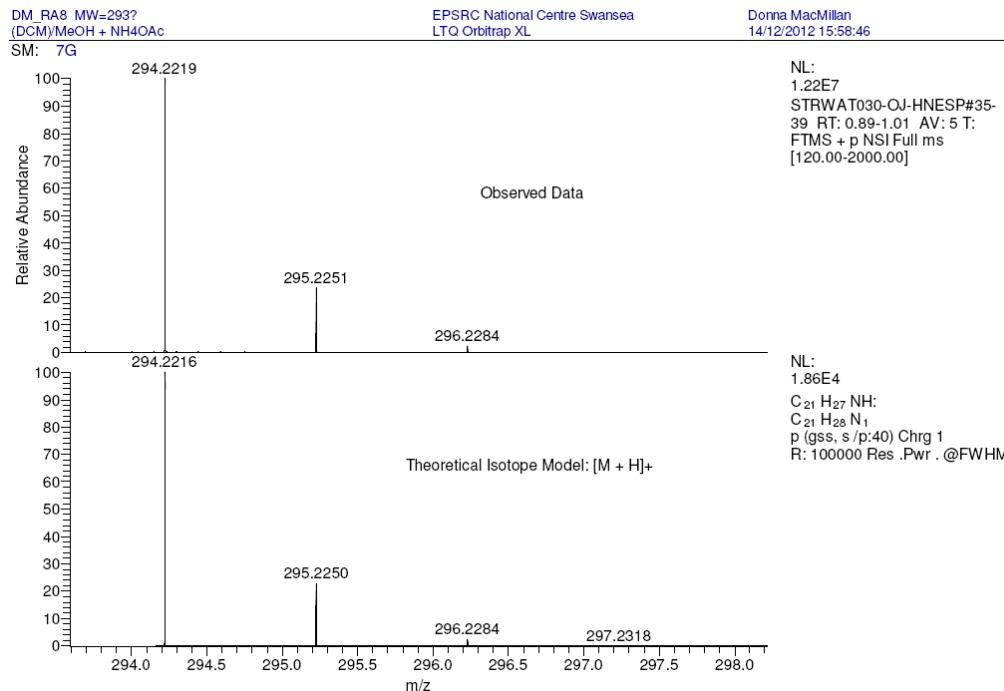
^{13}C NMR:



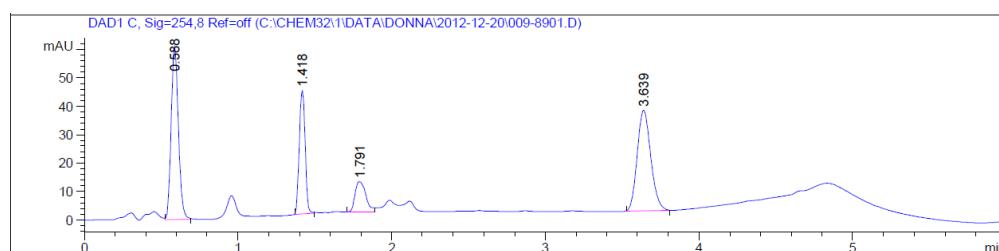
FTIR:



HRMS:



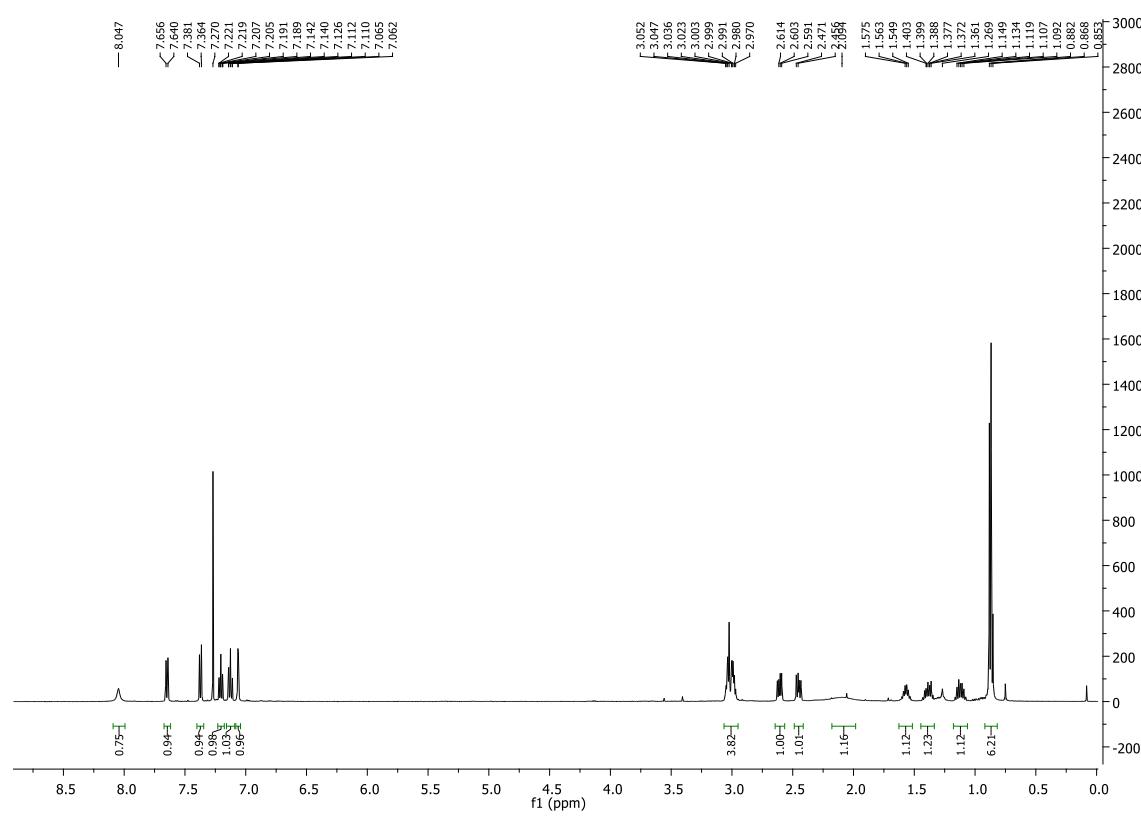
HPLC assay:



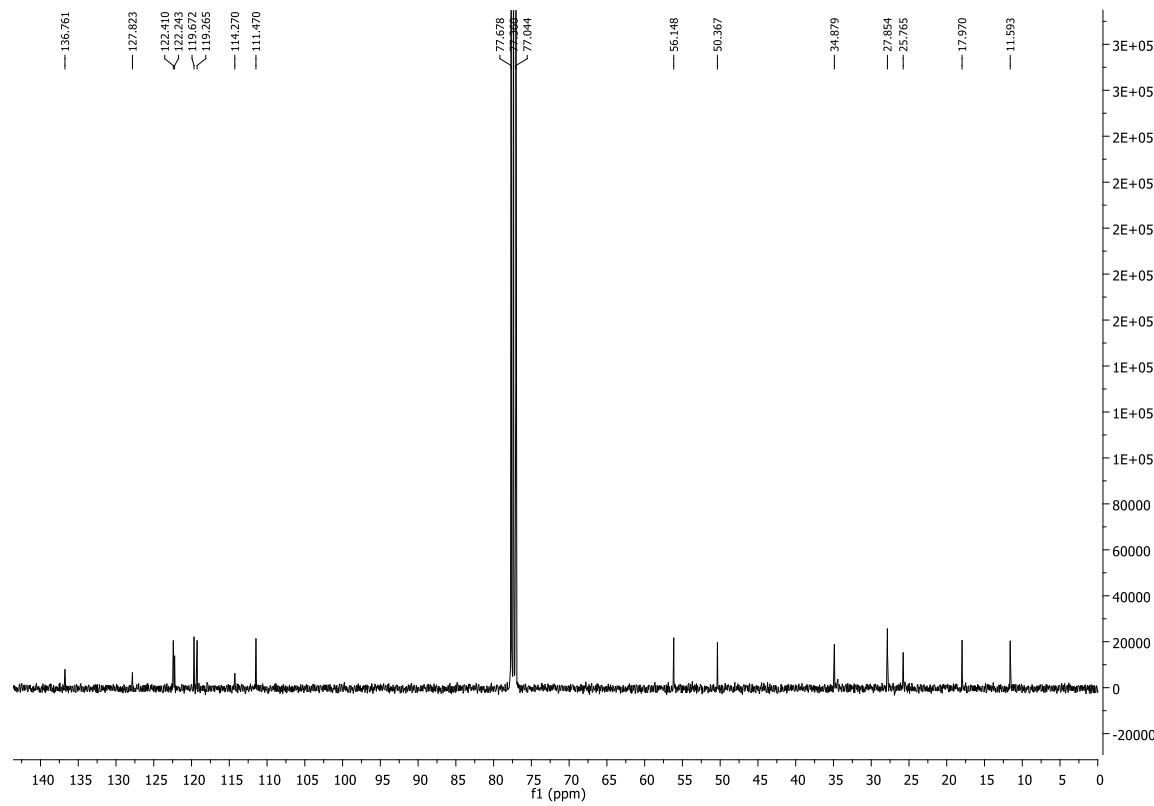
Substrate	R _t (min)
Compound 8: 4-Benzyl-1-(3-phenylpropyl)piperidine	1.42
Iodobenzene (standard)	3.64

Compound 13: N-(2-(1H-Indol-3-yl)ethyl)-2-methylbutan-1-amine

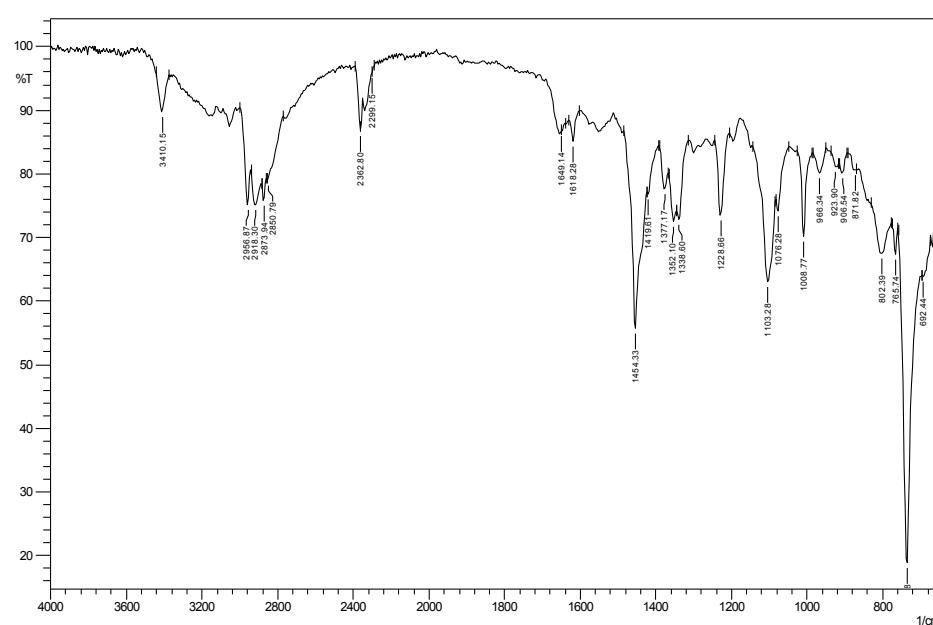
^1H NMR:



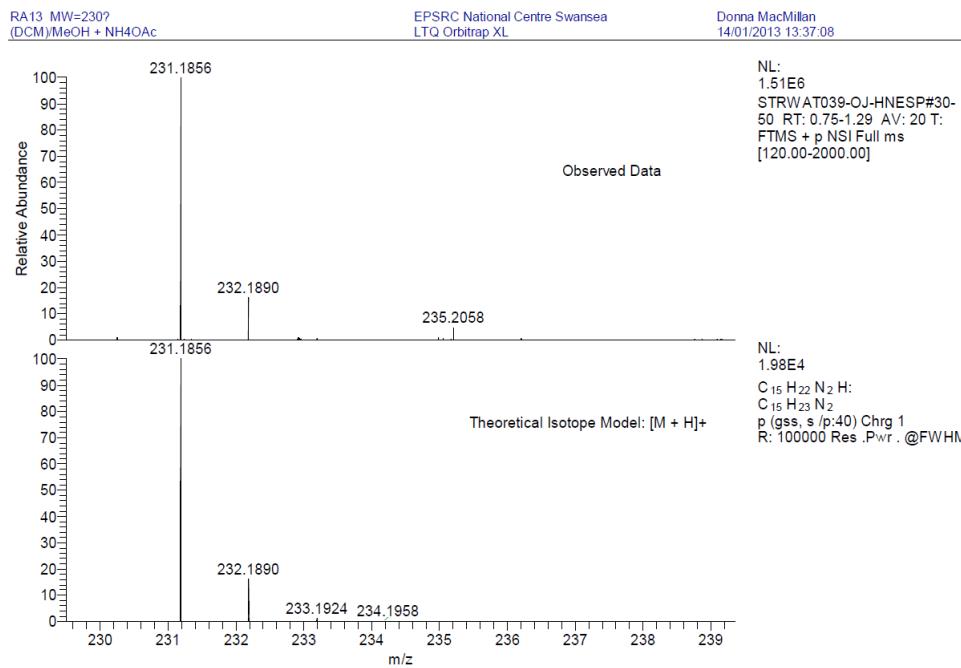
^{13}C NMR:



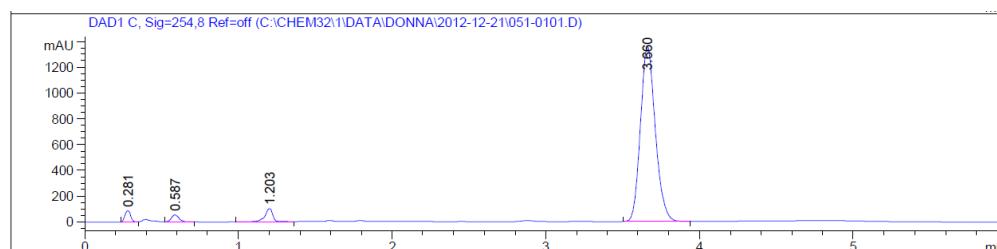
FTIR:



HRMS:



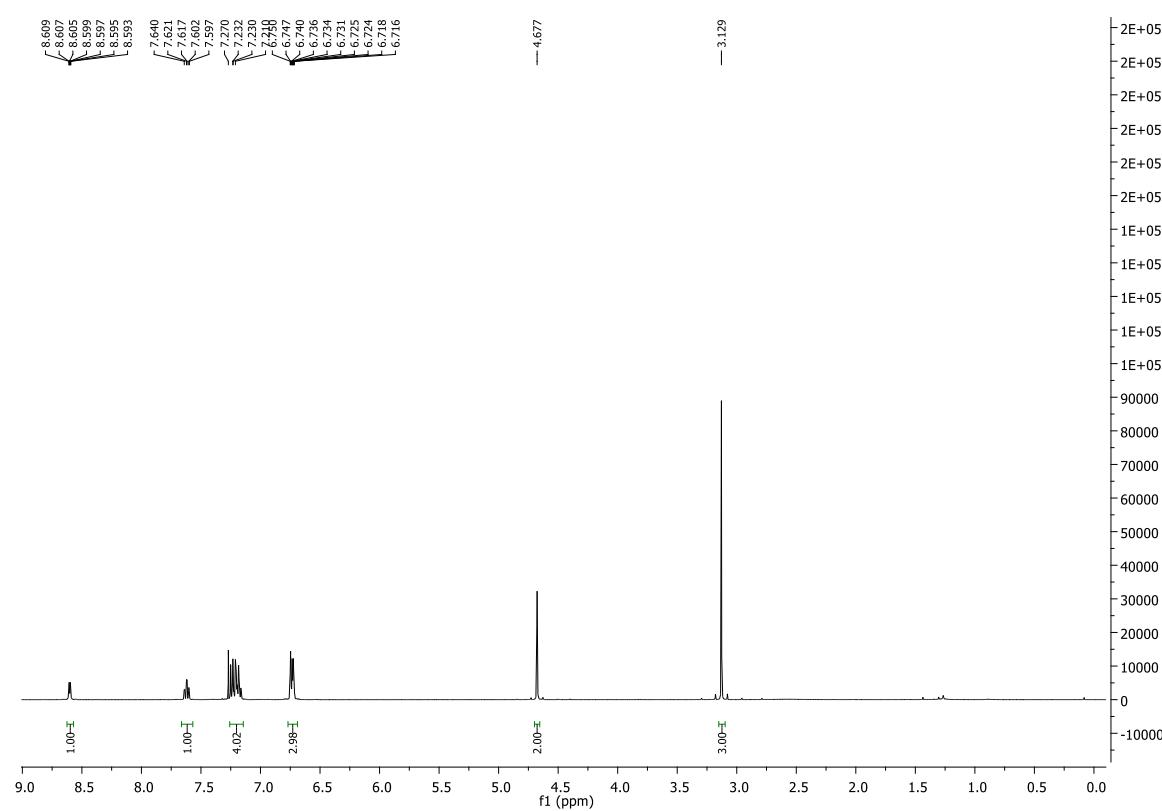
HPLC assay:



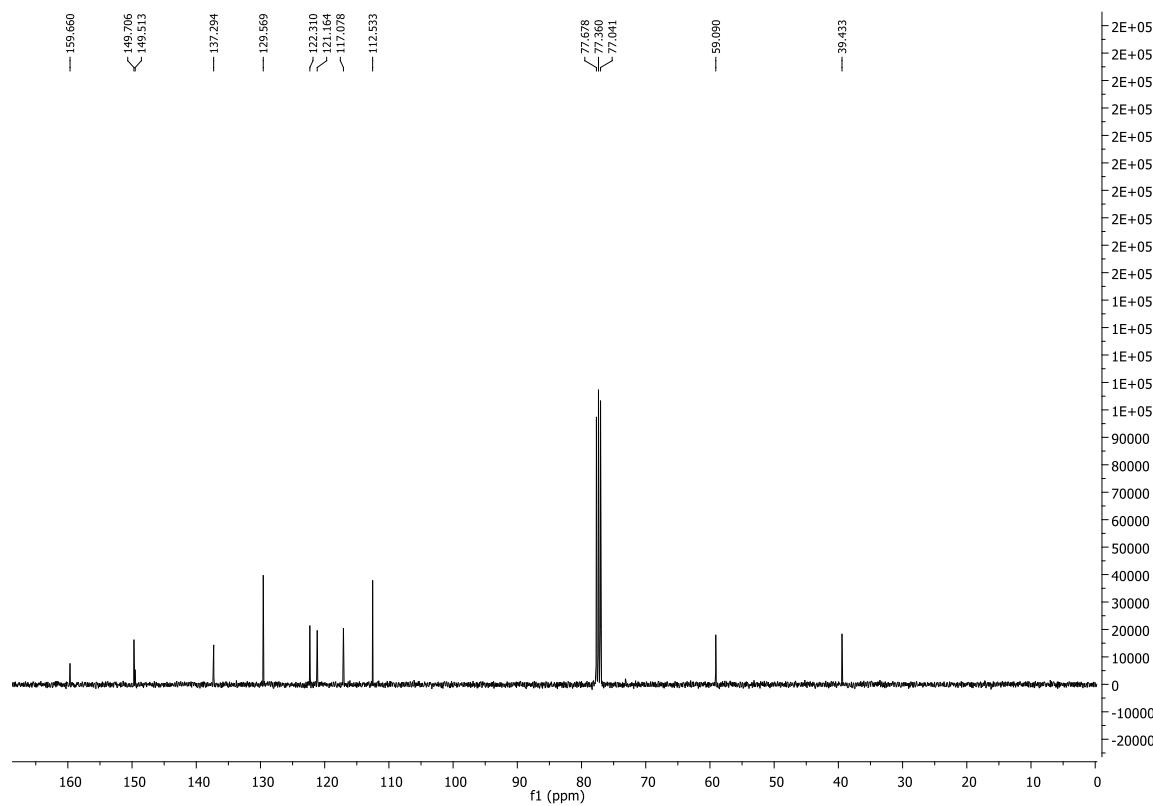
Substrate	R _t (min)
Compound 13: N-(2-(1 <i>H</i> -indol-3-yl)ethyl)-2-methylbutan-1-amine	1.20
Iodobenzene (standard)	3.66

Compound 14: N-Methyl-N-(pyridin-2-ylmethyl)aniline

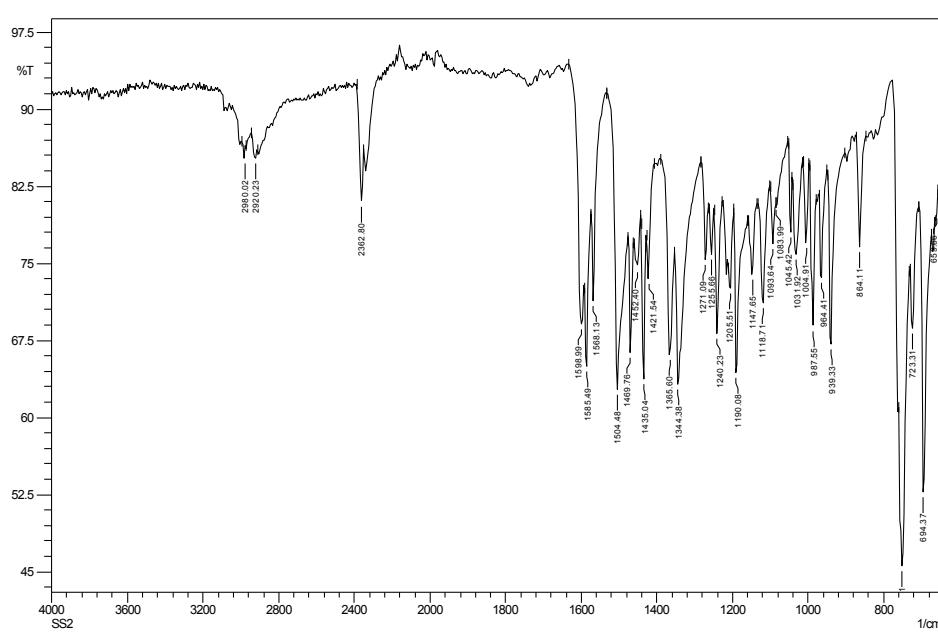
^1H NMR:



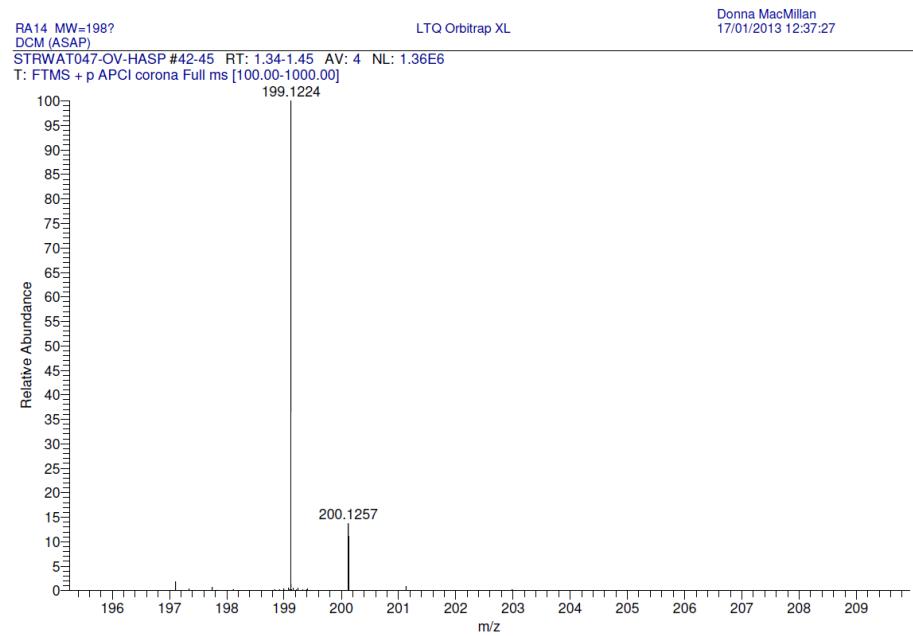
^{13}C NMR:



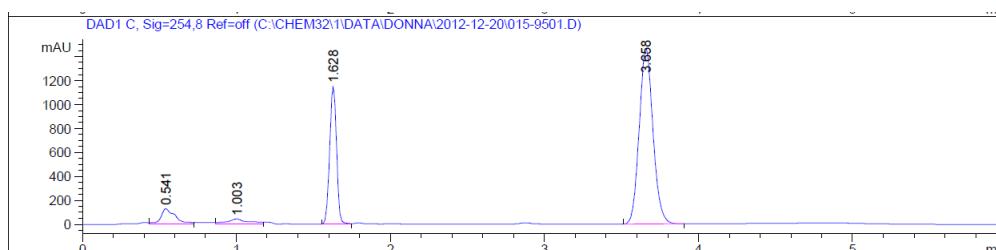
FTIR:



HRMS:



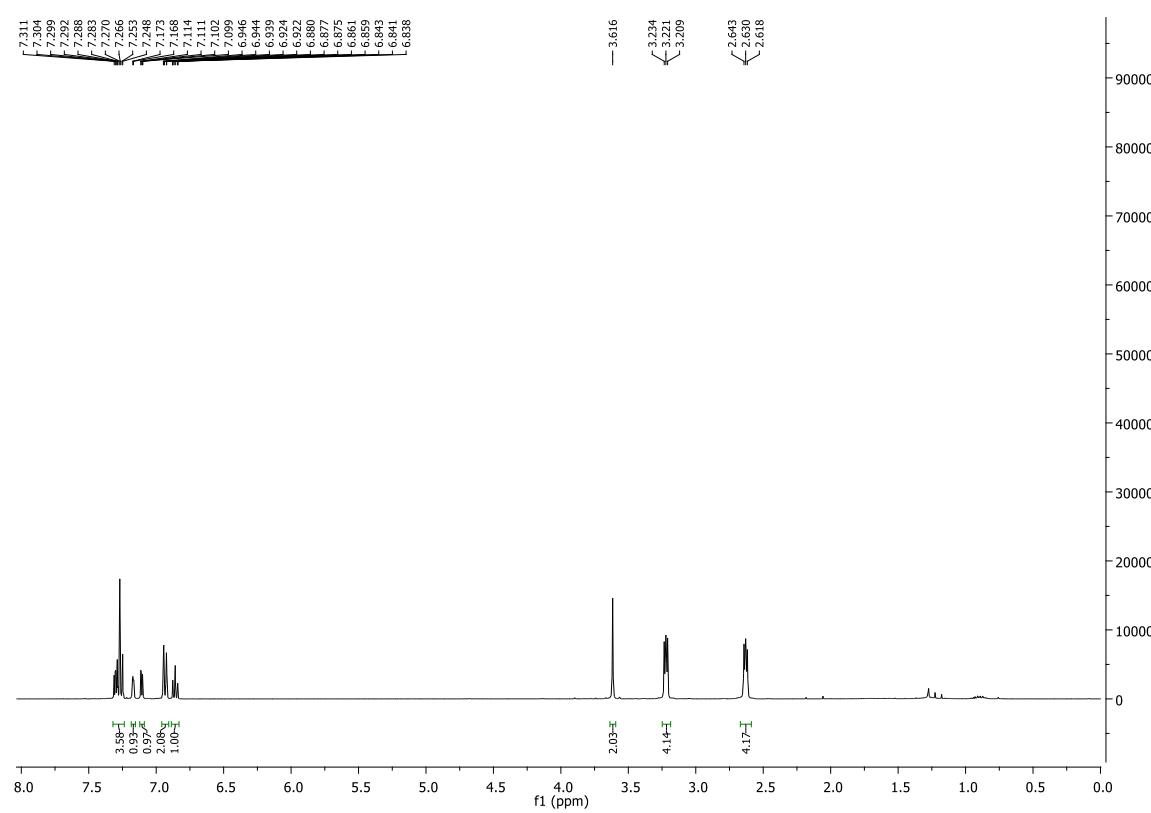
HPLC assay:



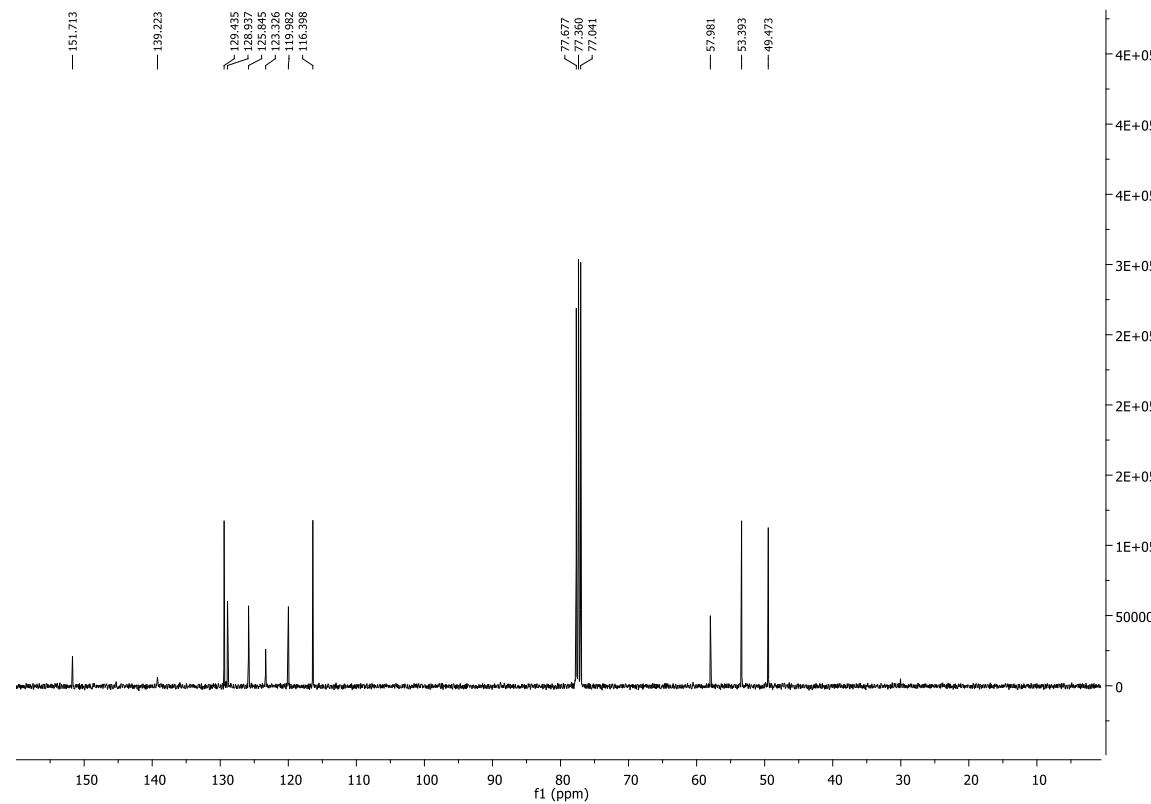
Substrate	R _t (min)
Compound 14: N-methyl-N-(pyridin-2-ylmethyl)aniline	1.63
Iodobenzene (standard)	3.66

Compound 15: 1-Phenyl-4-(thiophen-3-ylmethyl)piperazine

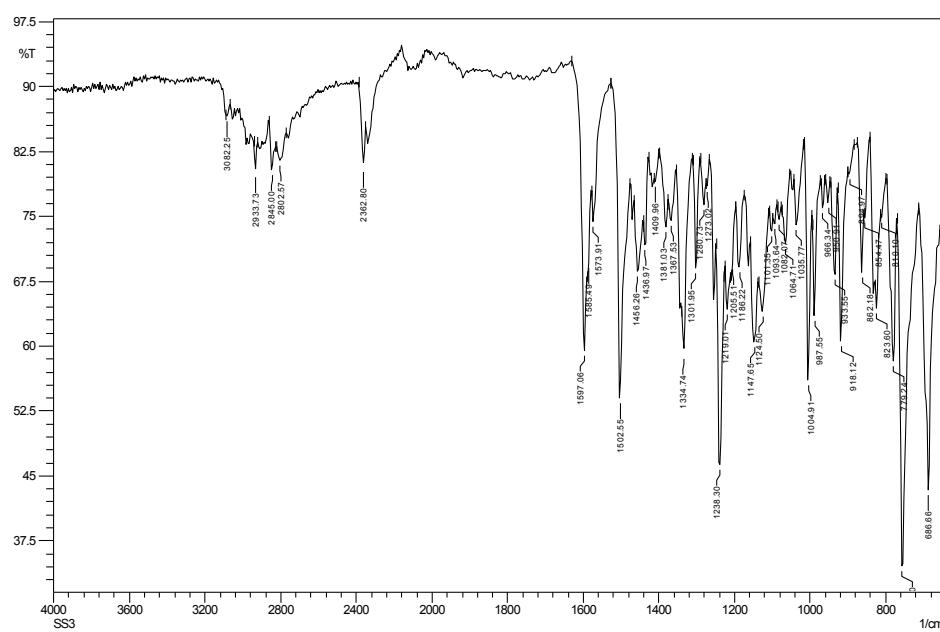
^1H NMR:



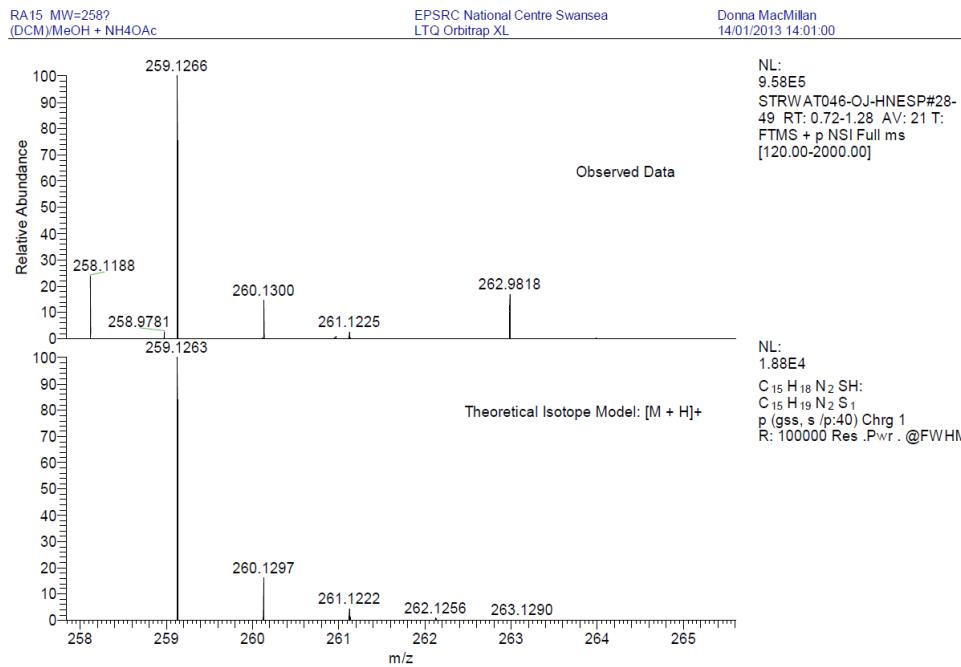
^{13}C NMR:



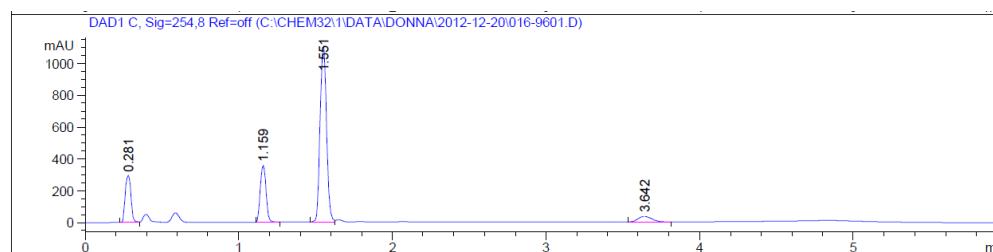
FTIR:



HRMS:



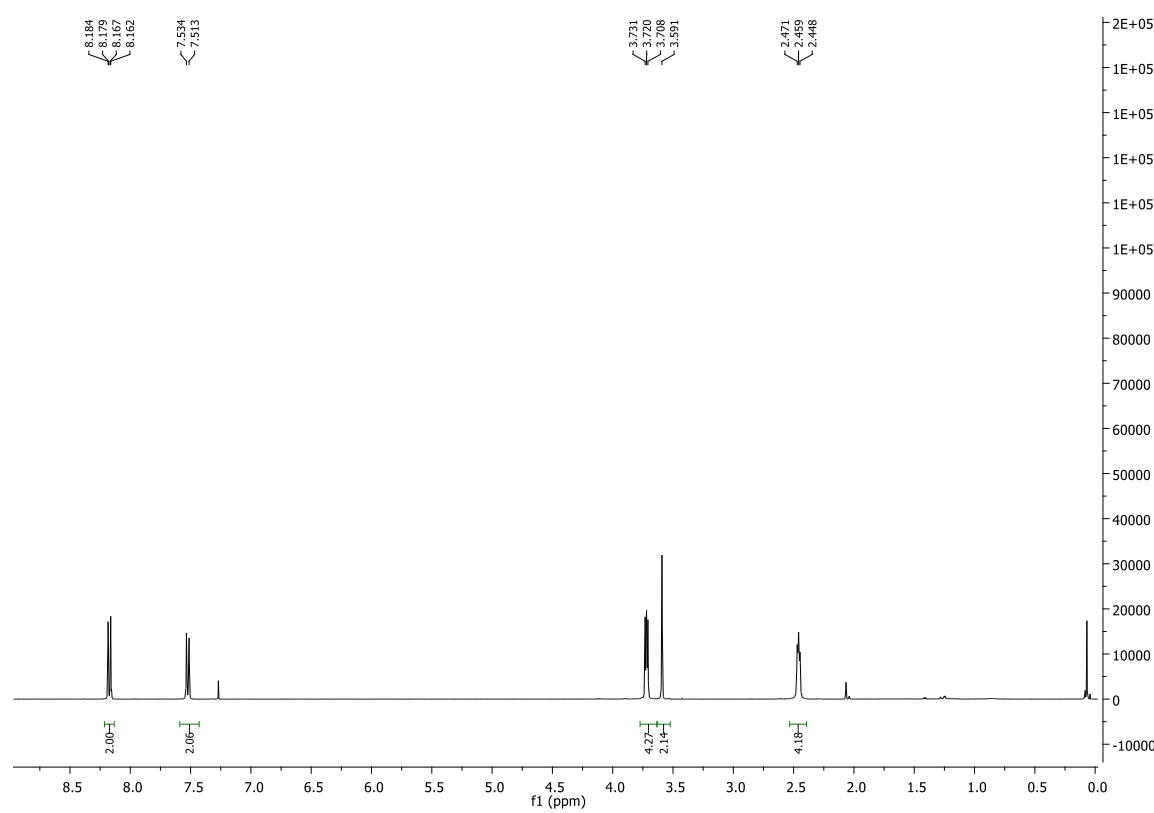
HPLC assay:



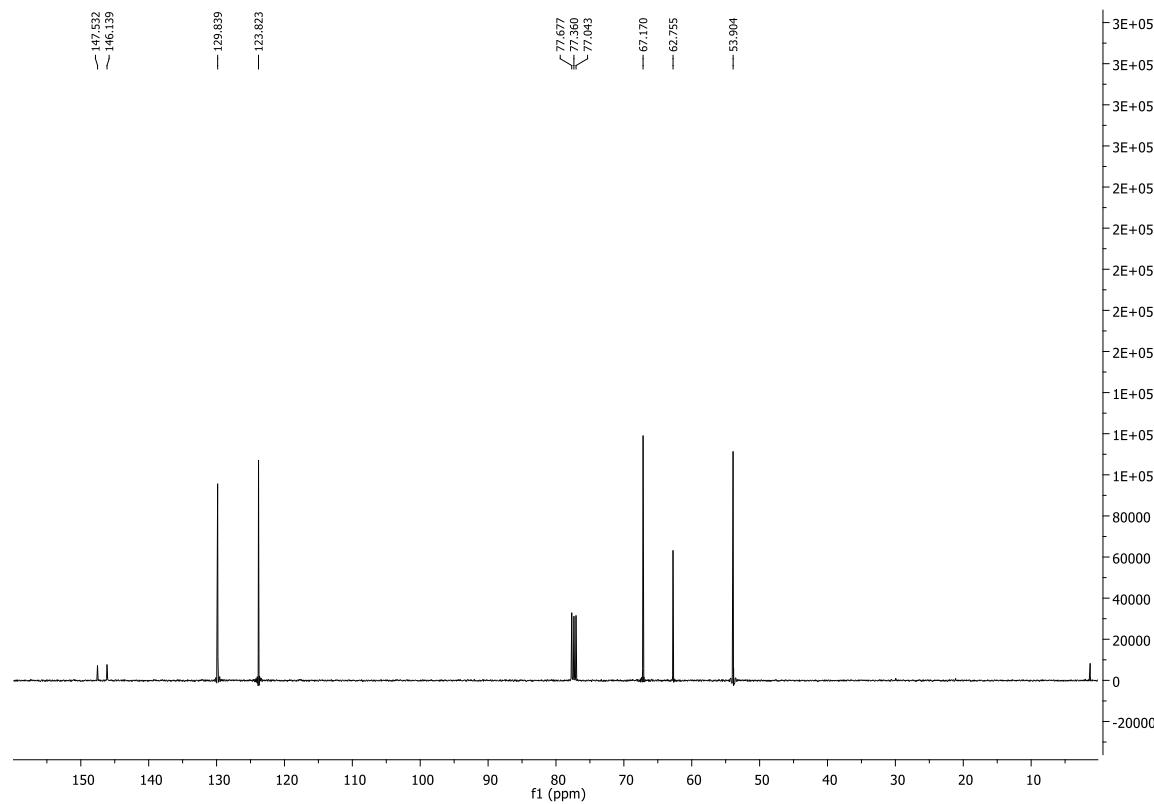
Substrate	R _t (min)
Compound 15: 1-Phenyl-4-(thiophen-3-ylmethyl)piperazine	1.56
Iodobenzene (standard)	3.64

Compound 16: 4-(4-Nitrobenzyl)morpholine

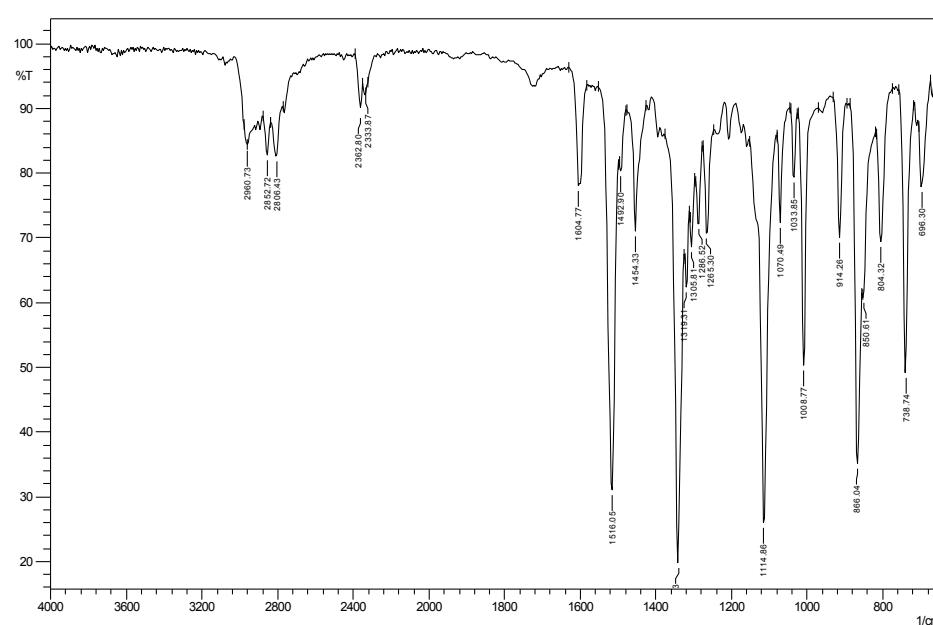
^1H NMR:



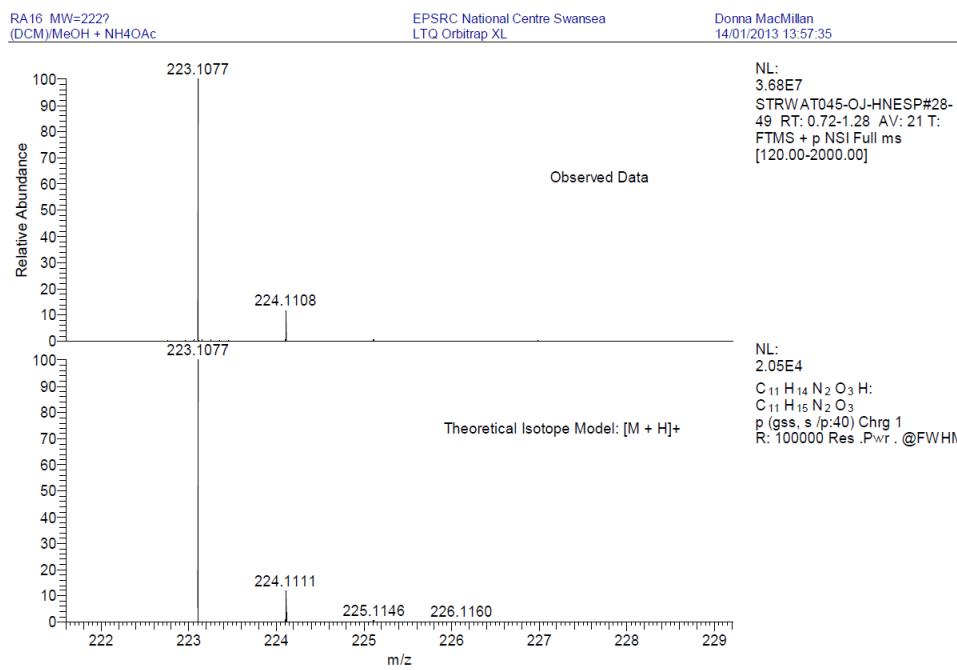
^{13}C NMR:



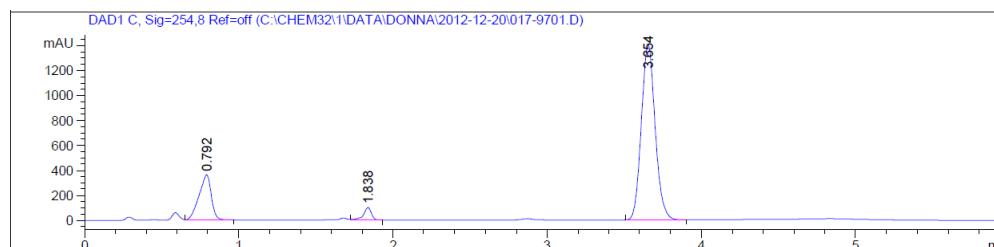
FTIR:



HRMS:



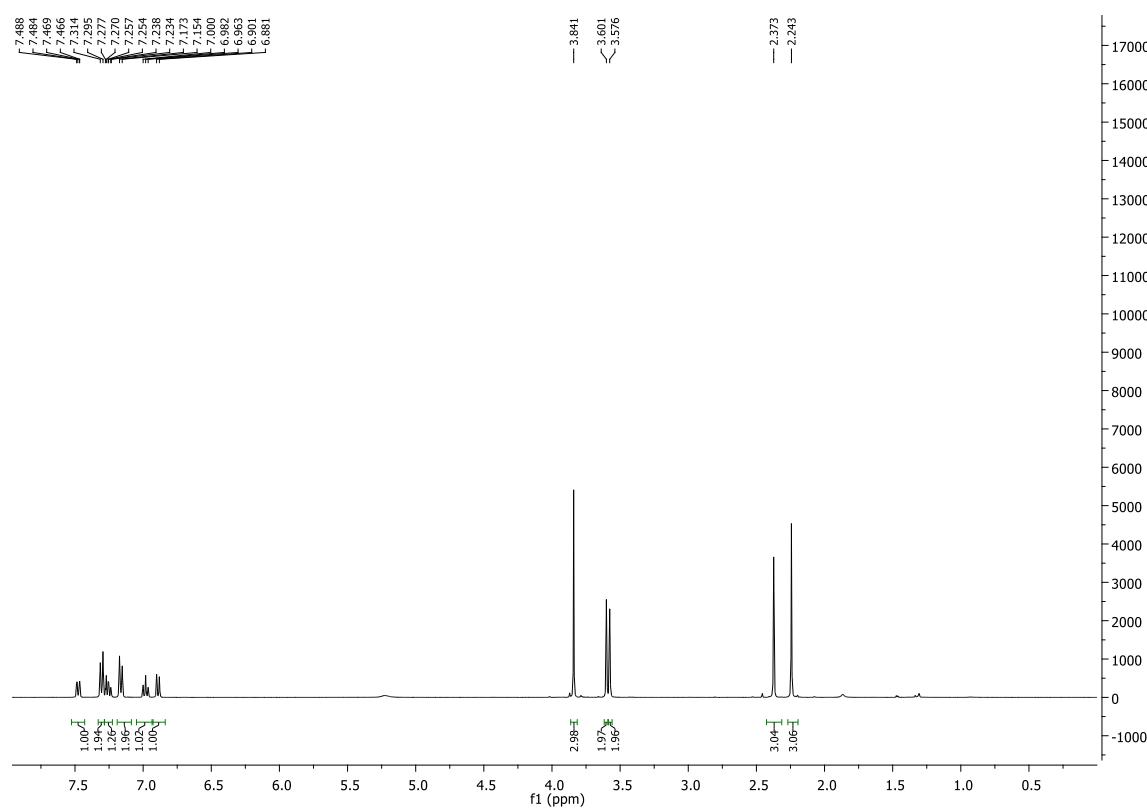
HPLC assay:



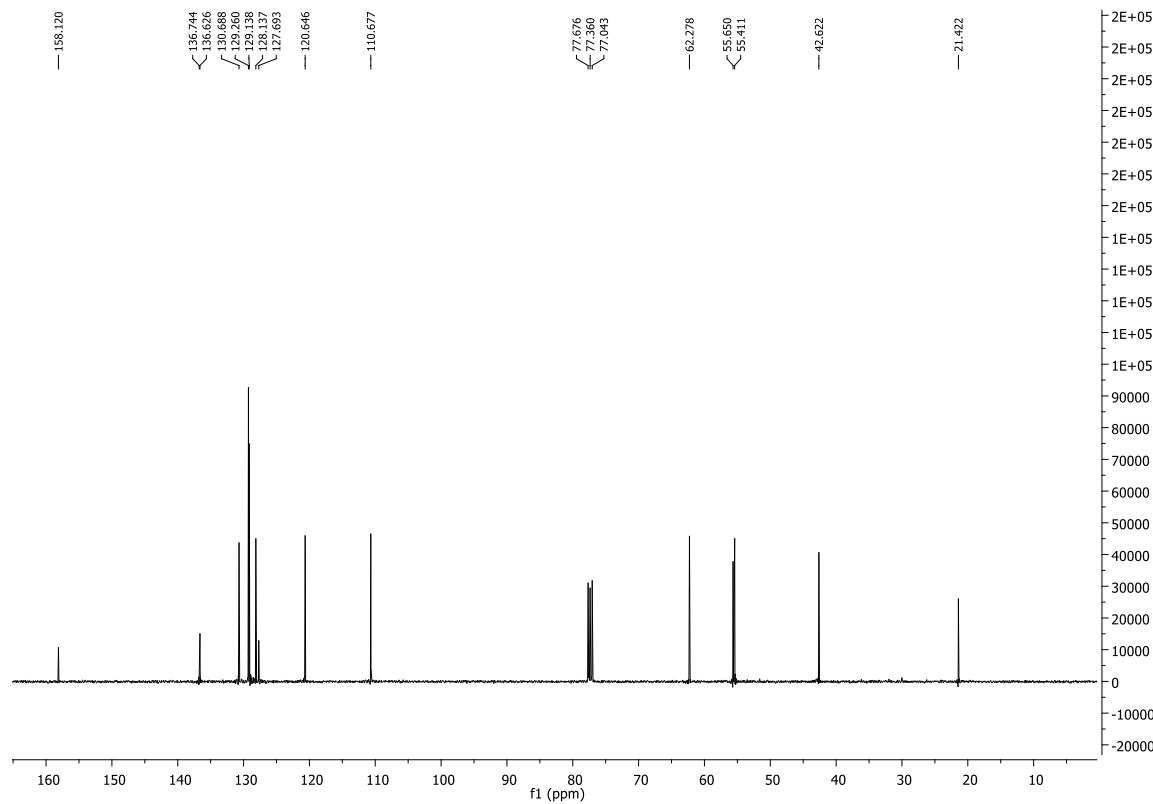
Substrate	R _t (min)
Compound 16: 4-(4-Nitrobenzyl)morpholine	0.79
Iodobenzene (standard)	3.65

Compound 17: N-(3-Methoxybenzyl)-N-methyl-1-(*p*-tolyl)methanamine

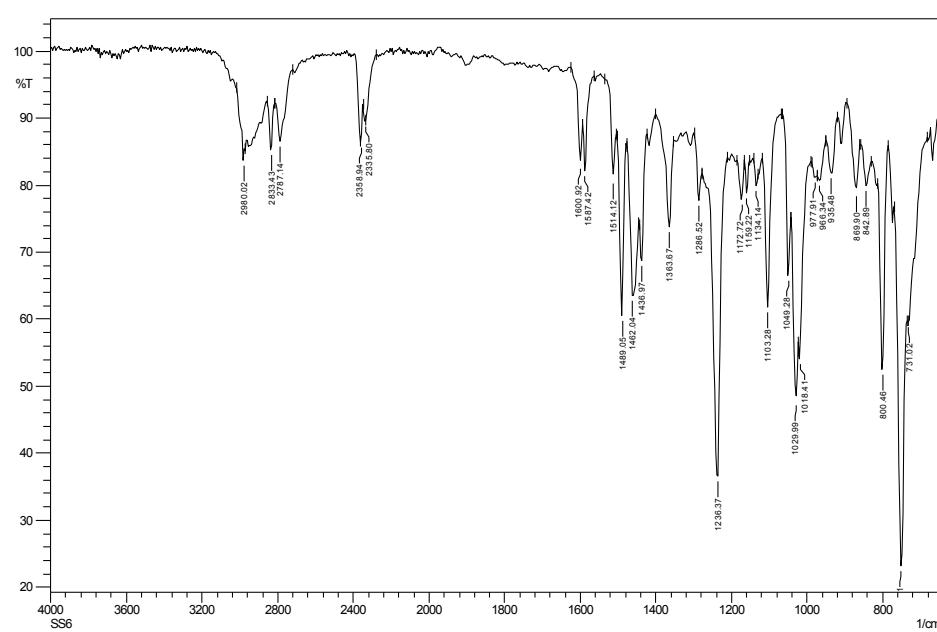
^1H NMR:



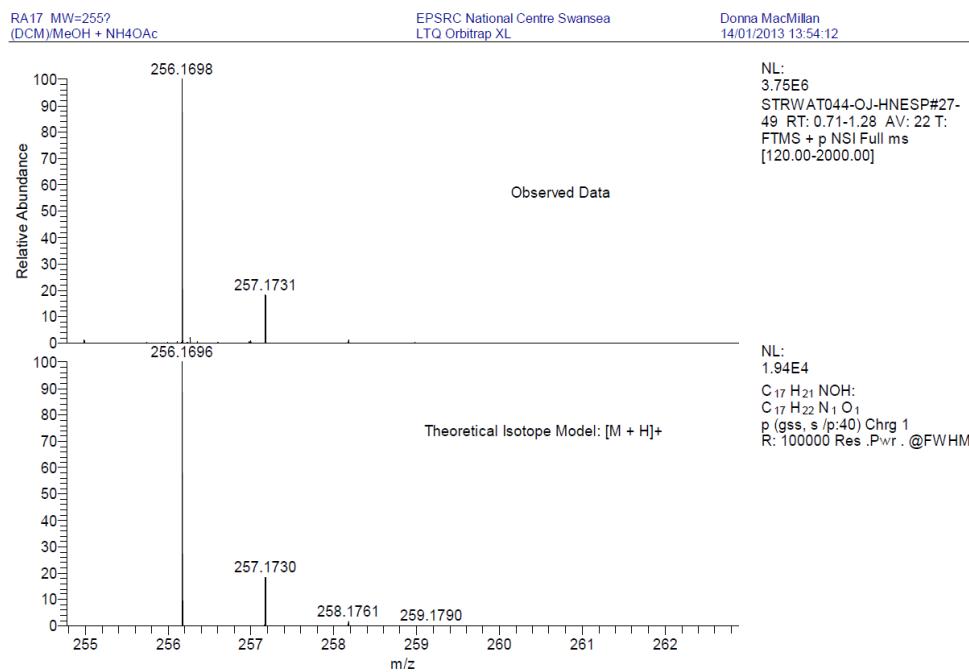
^{13}C NMR:



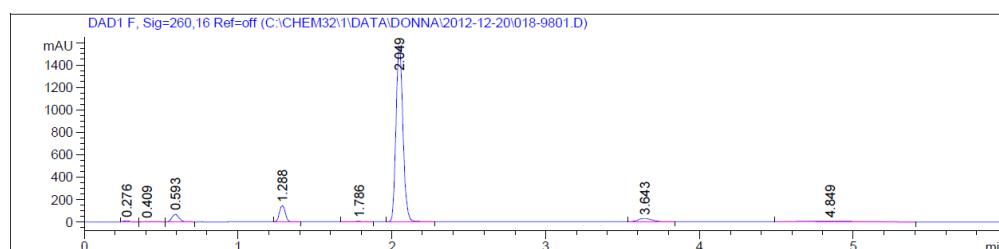
FTIR:



HRMS:



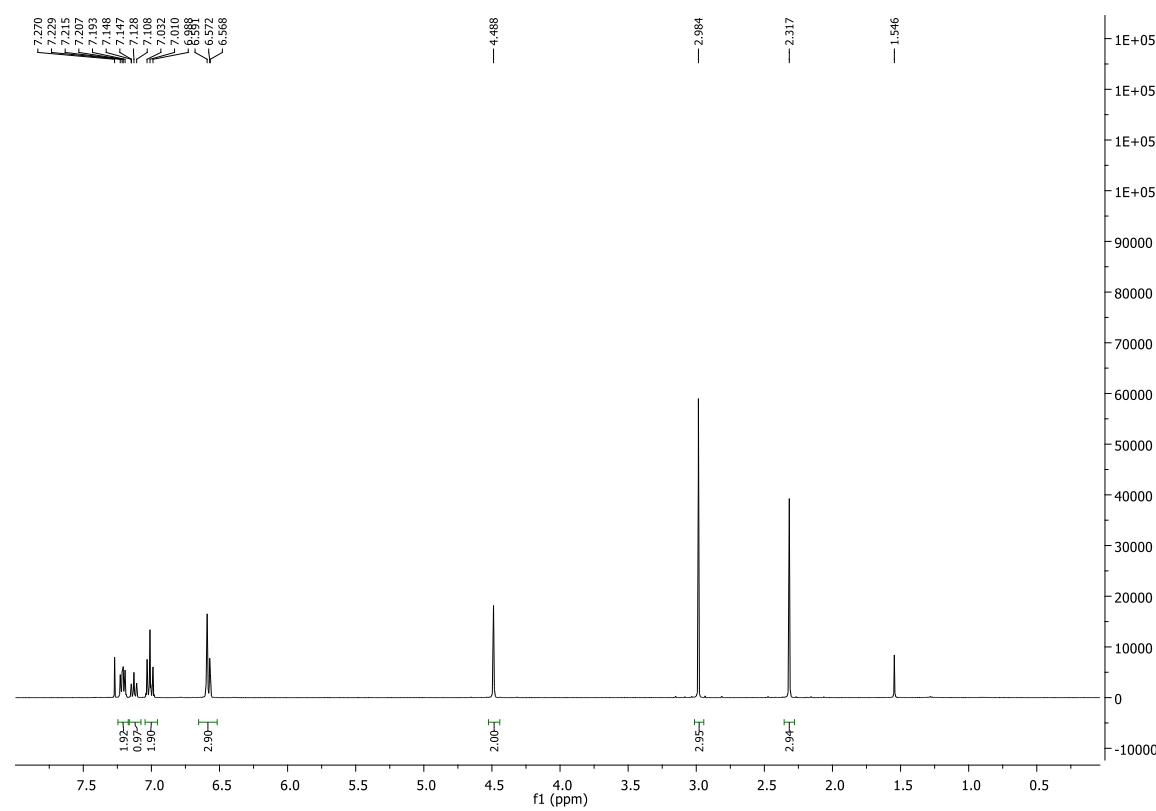
HPLC assay:



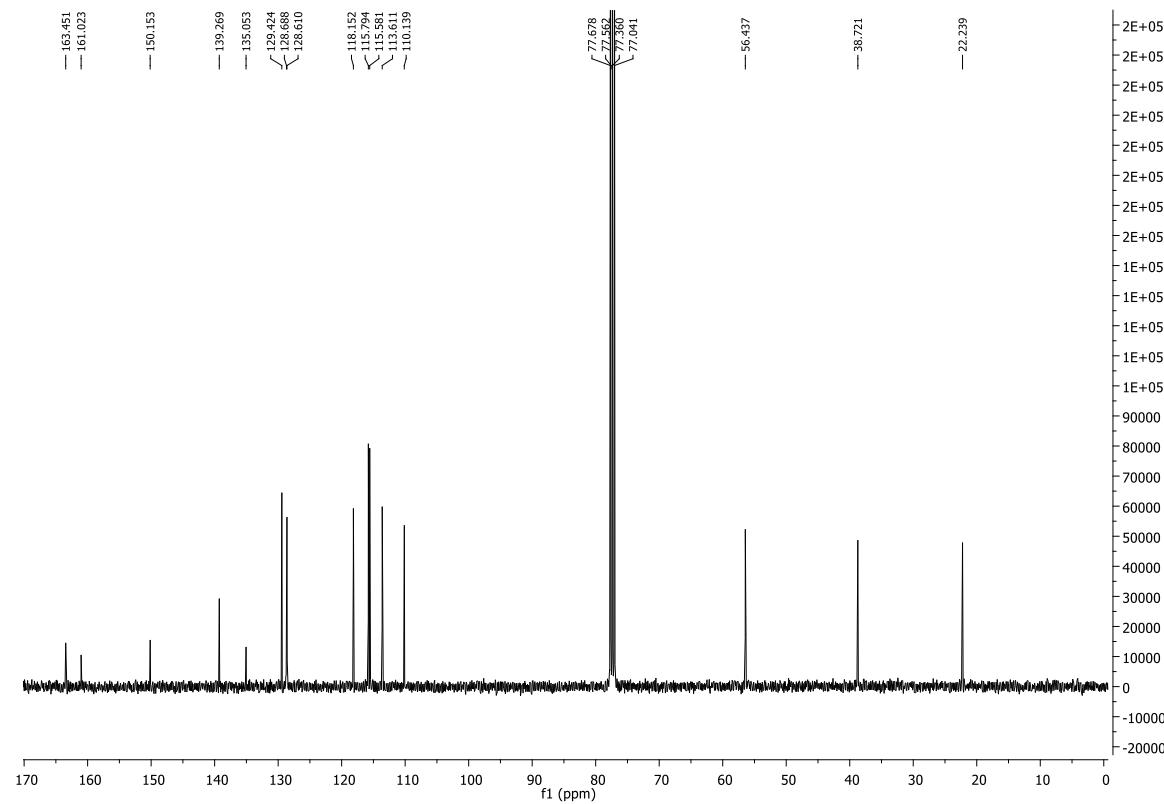
Substrate	R _t (min)
Compound 17: N-(3-methoxybenzyl)-N-methyl-1-(p-tolyl)methanamine	2.05
Iodobenzene (standard)	3.65

Compound 18: *N*-(4-Fluorobenzyl)-*N*,3-dimethylaniline

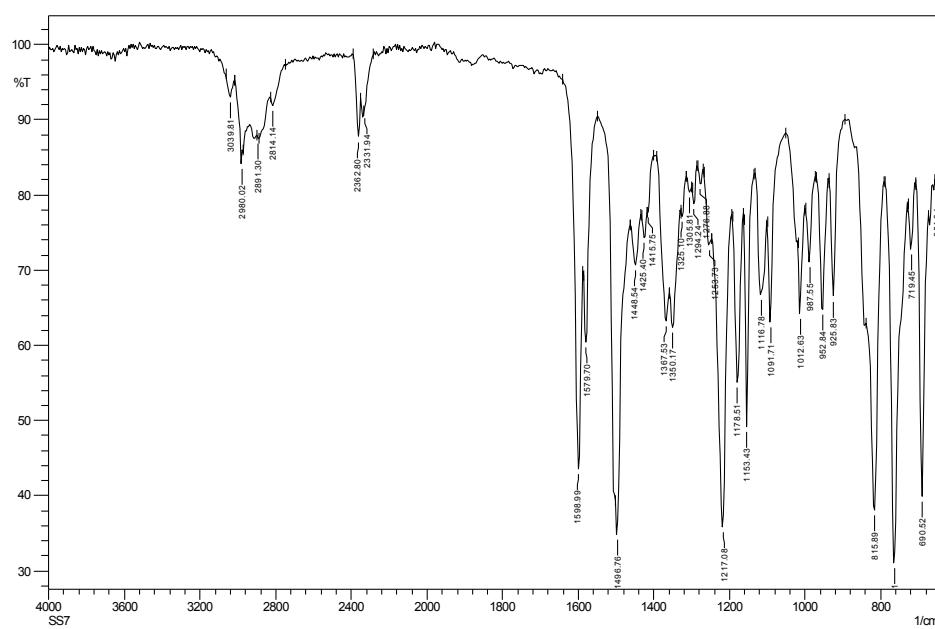
^1H NMR:



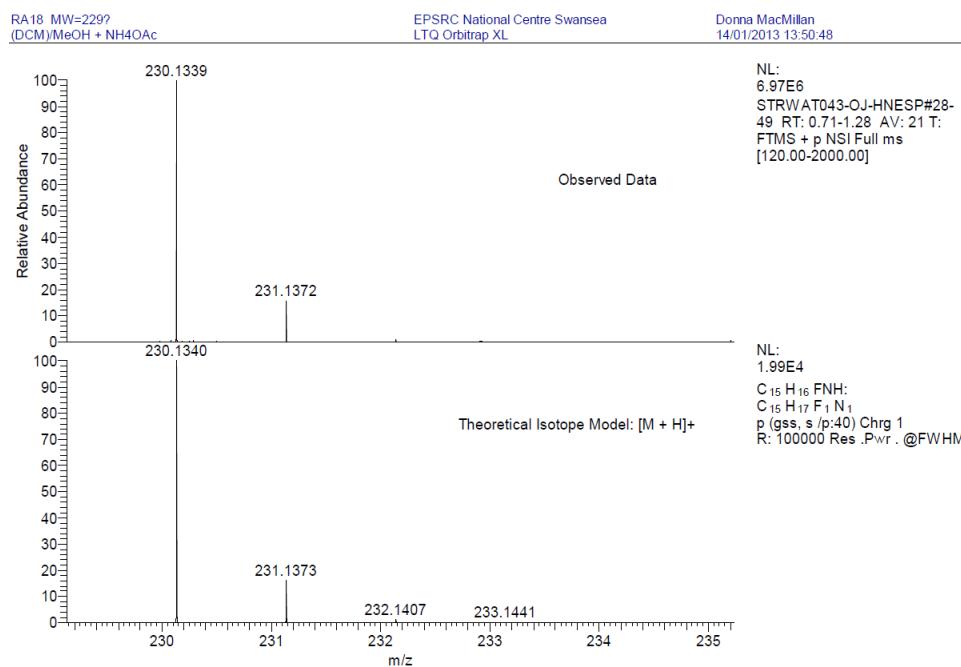
^{13}C NMR:



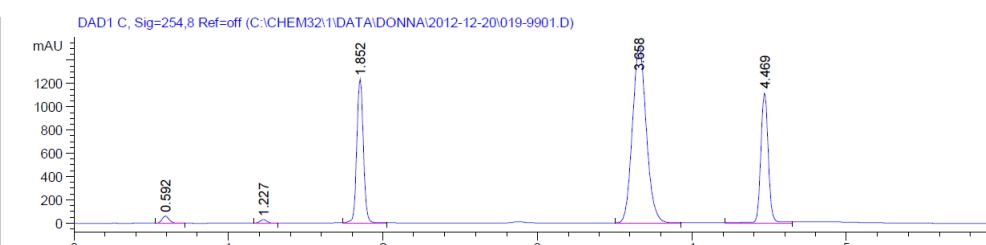
FTIR:



HRMS:



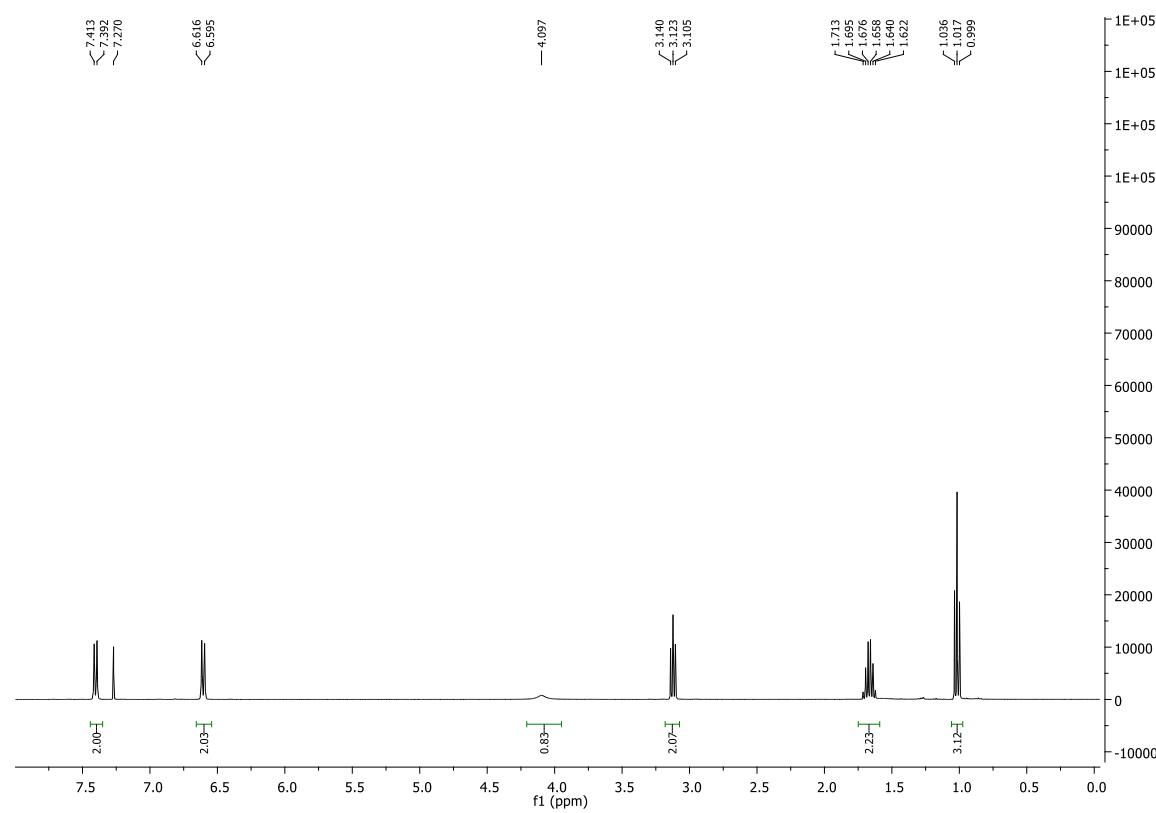
HPLC assay:



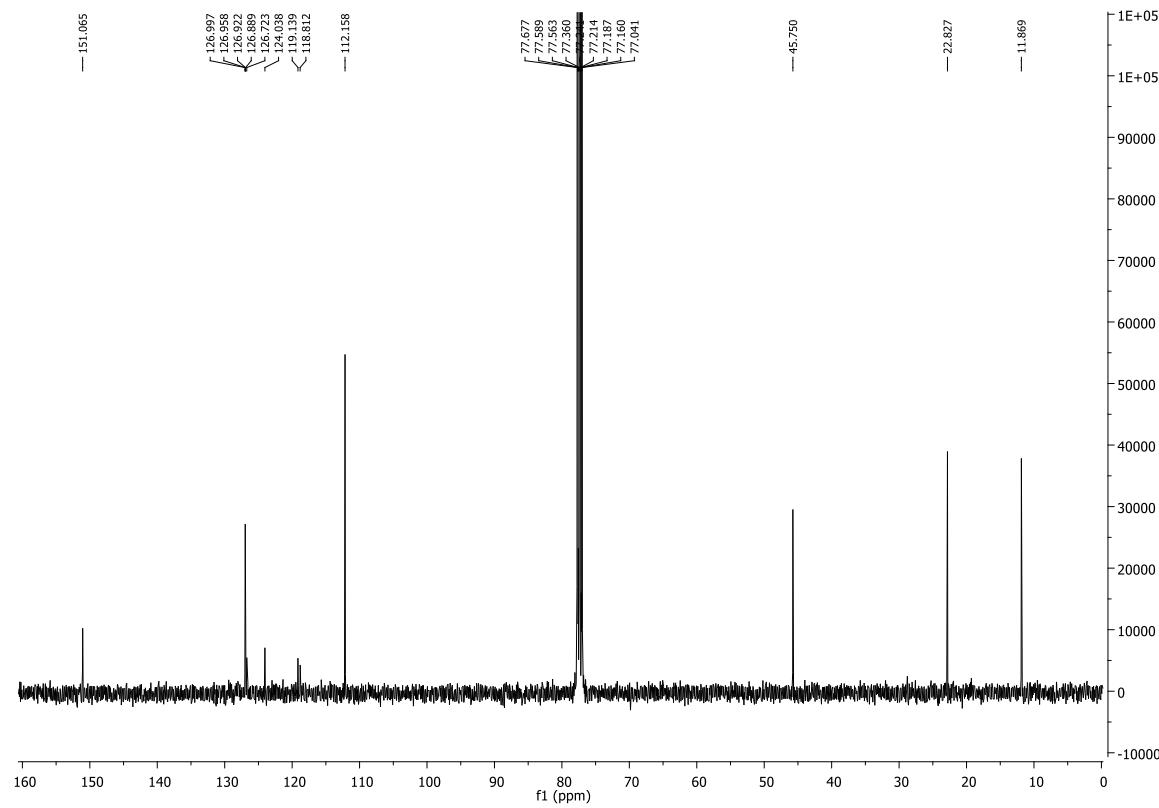
Substrate	R _t (min)
Compound 18: N-(4-fluorobenzyl)-N,3-dimethylaniline	4.46
Iodobenzene (standard)	3.65

Compound 19: N-Propyl-4-(trifluoromethyl)aniline

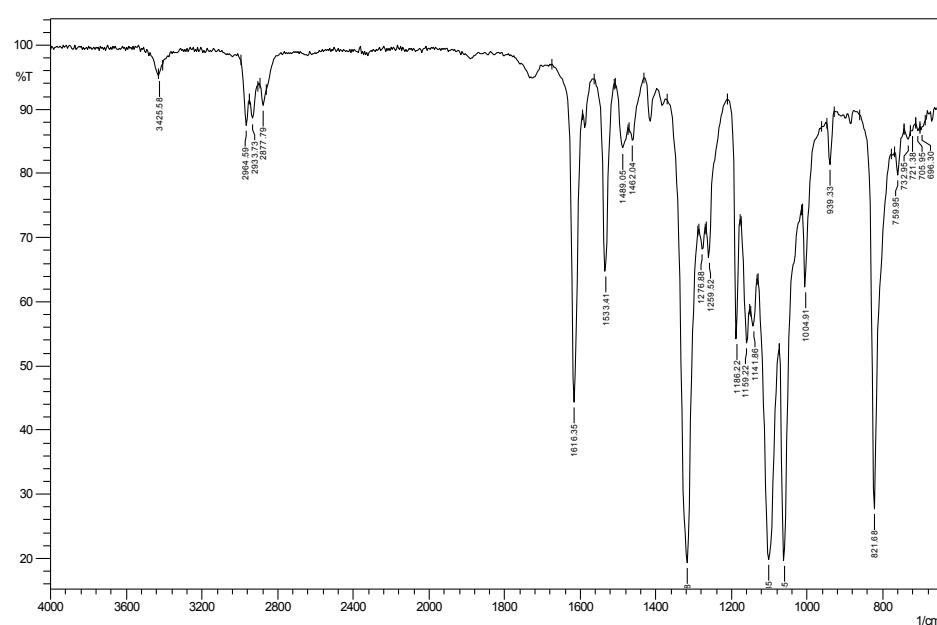
^1H NMR:



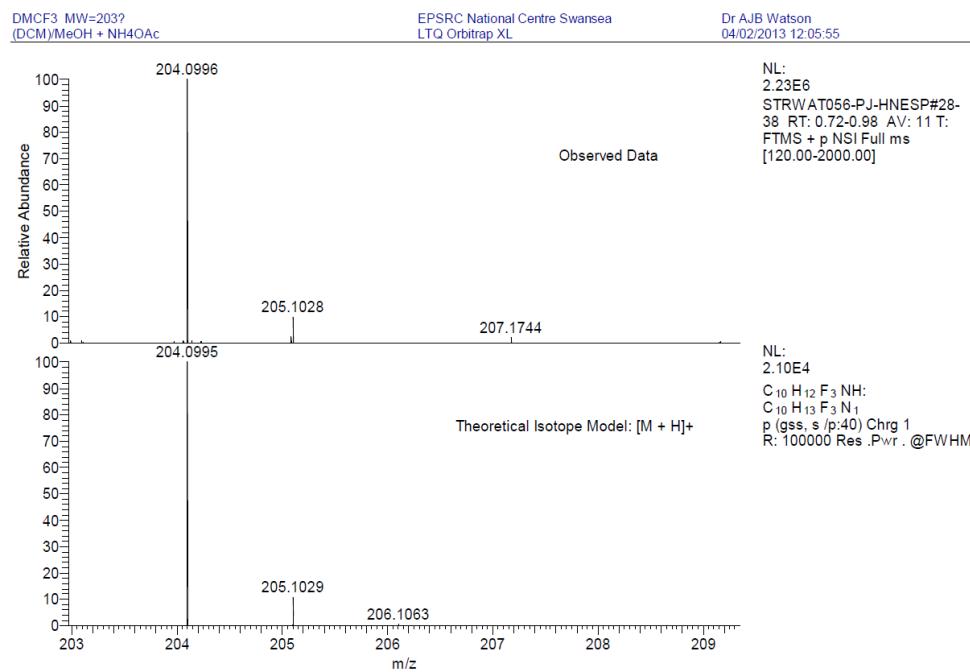
^{13}C NMR:



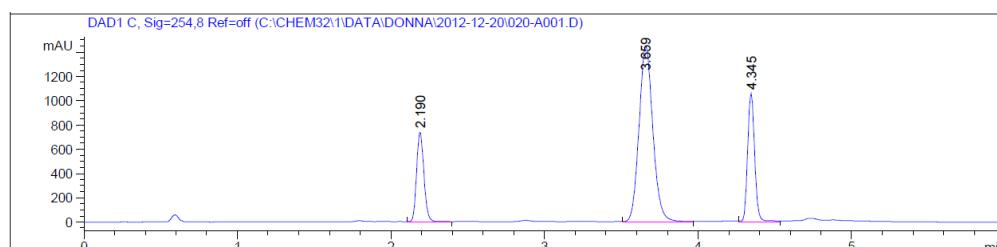
FTIR:



HRMS:



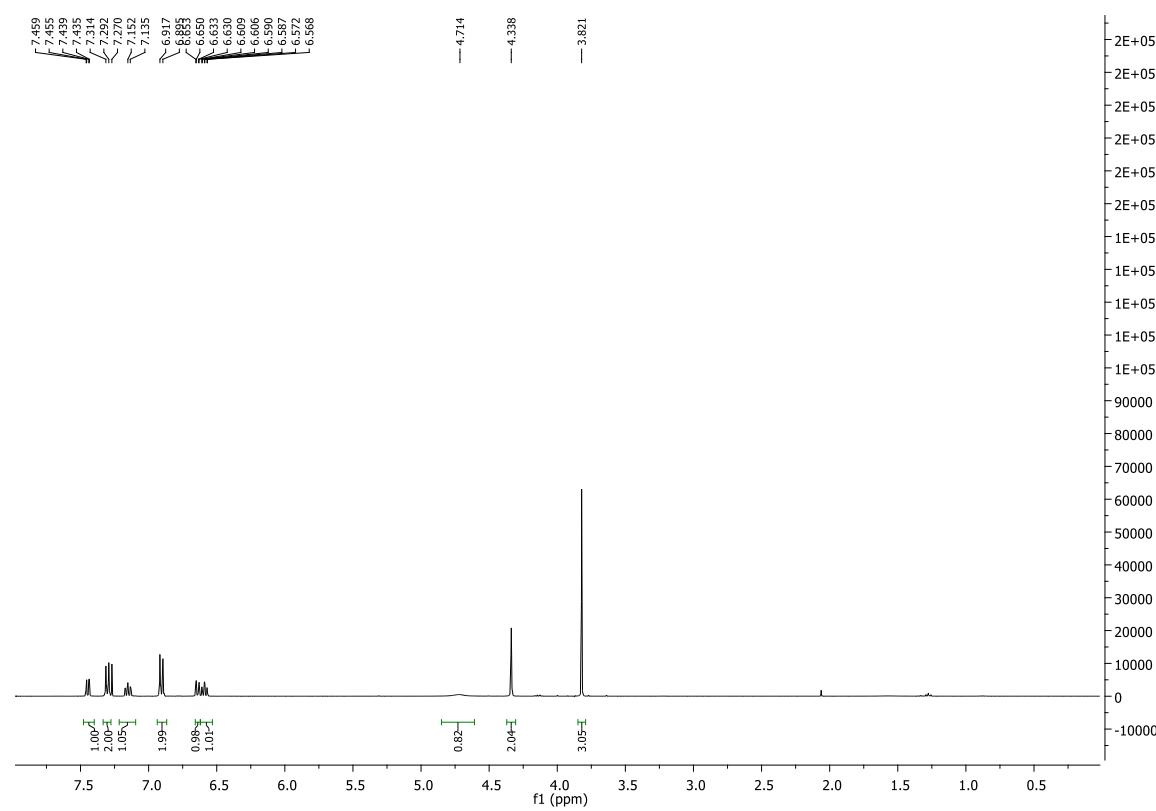
HPLC assay:



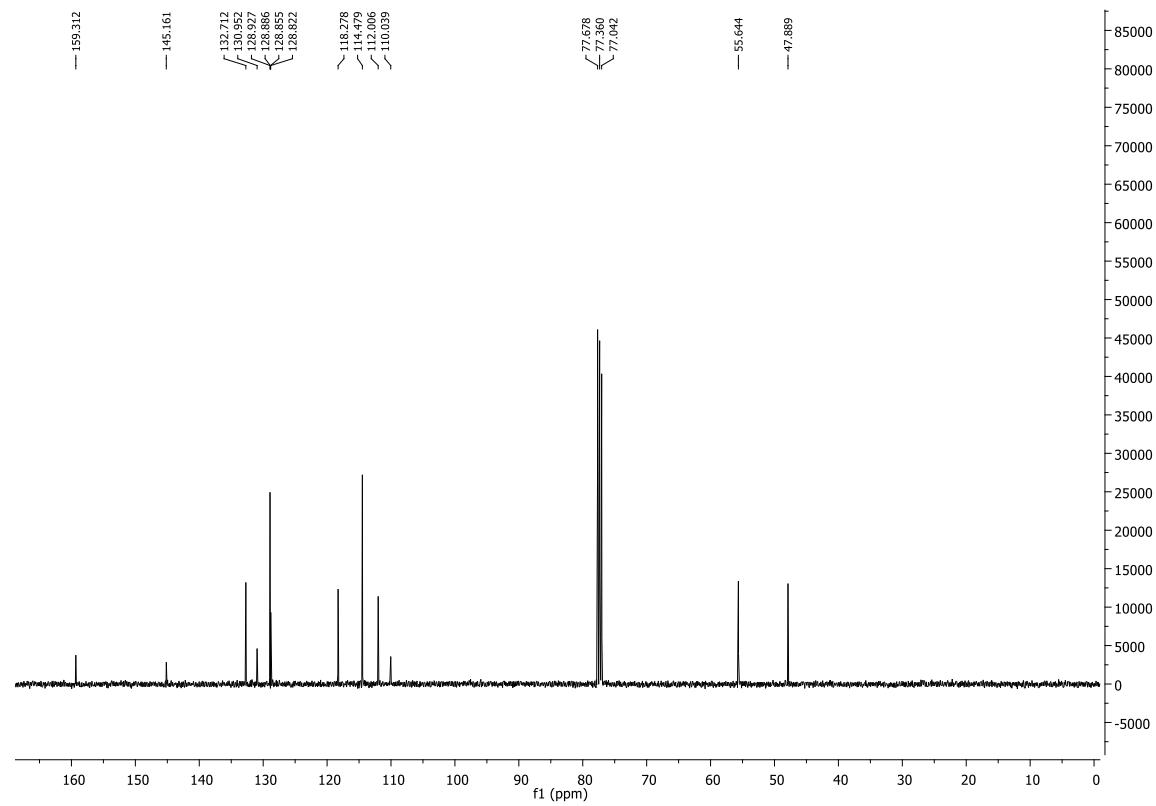
Substrate	R _t (min)
Compound 19: N-Propyl-4-(trifluoromethyl)aniline	4.35
Iodobenzene (standard)	3.65

Compound 20: 2-Bromo-N-(4-methoxybenzyl)aniline

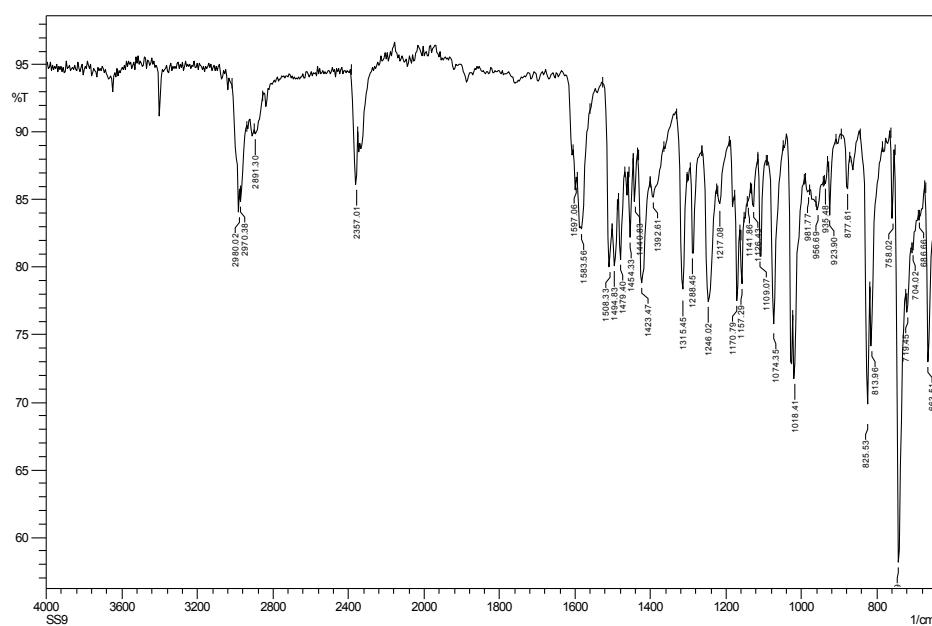
^1H NMR:



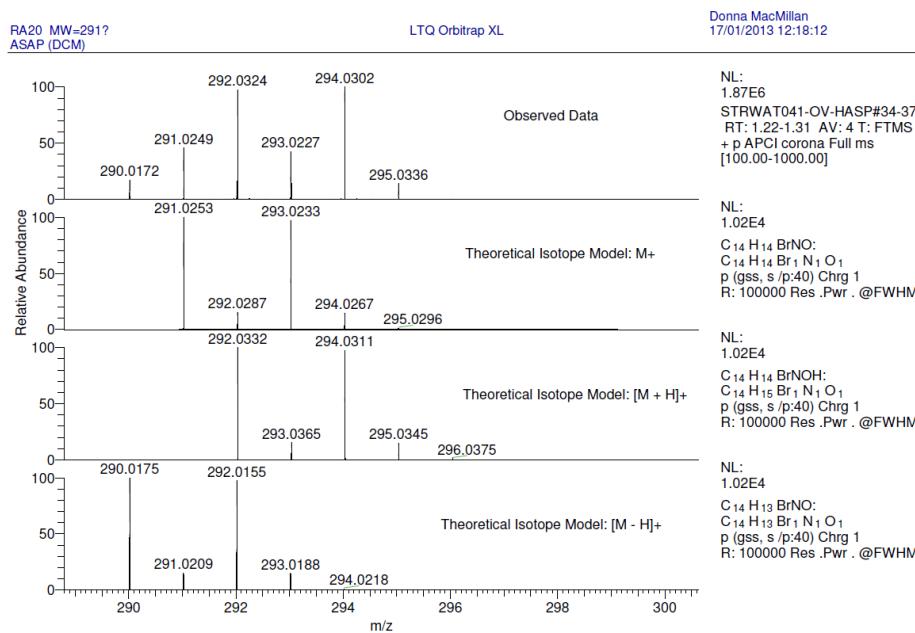
^{13}C NMR:



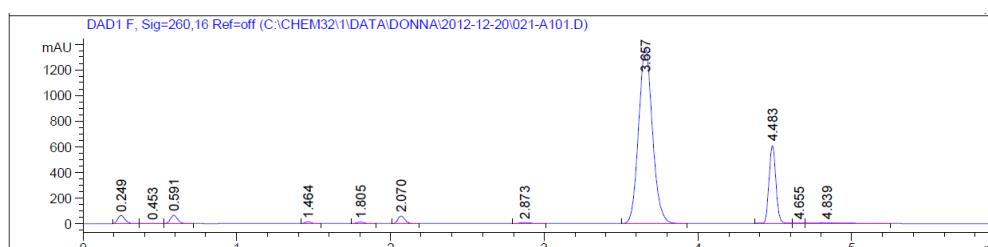
FTIR:



HRMS:



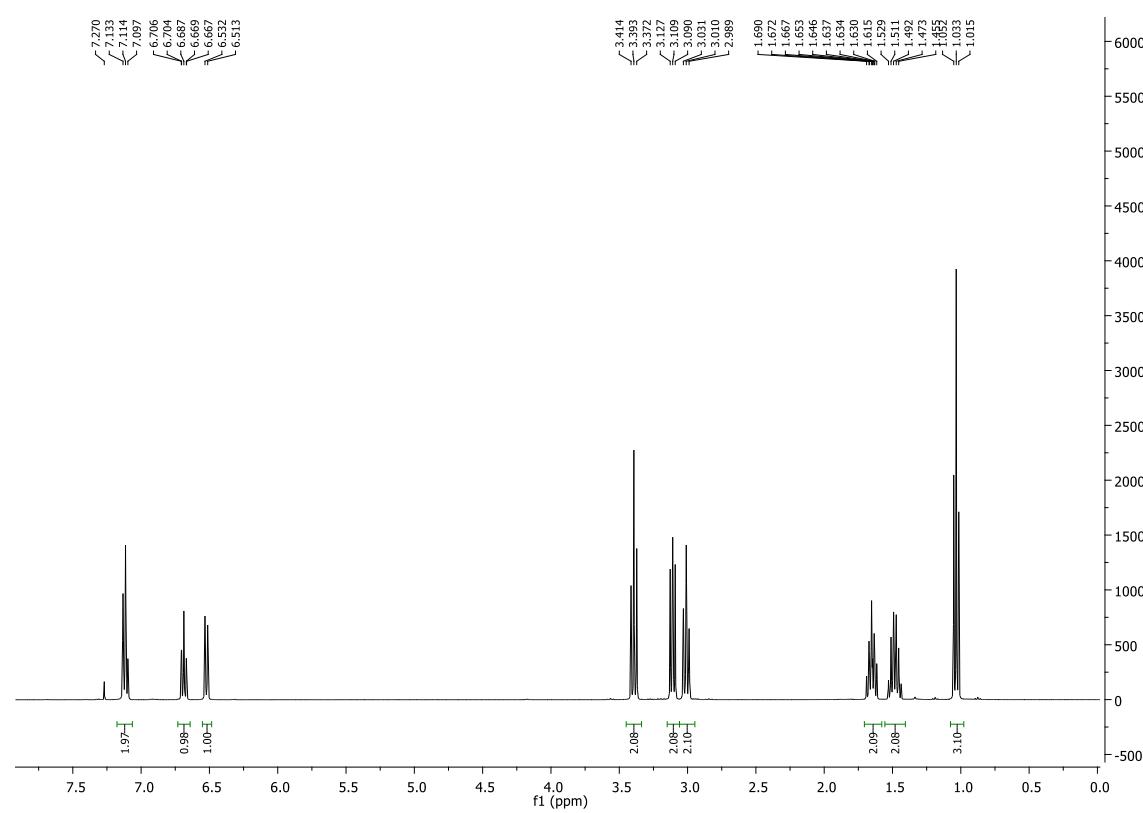
HPLC assay:



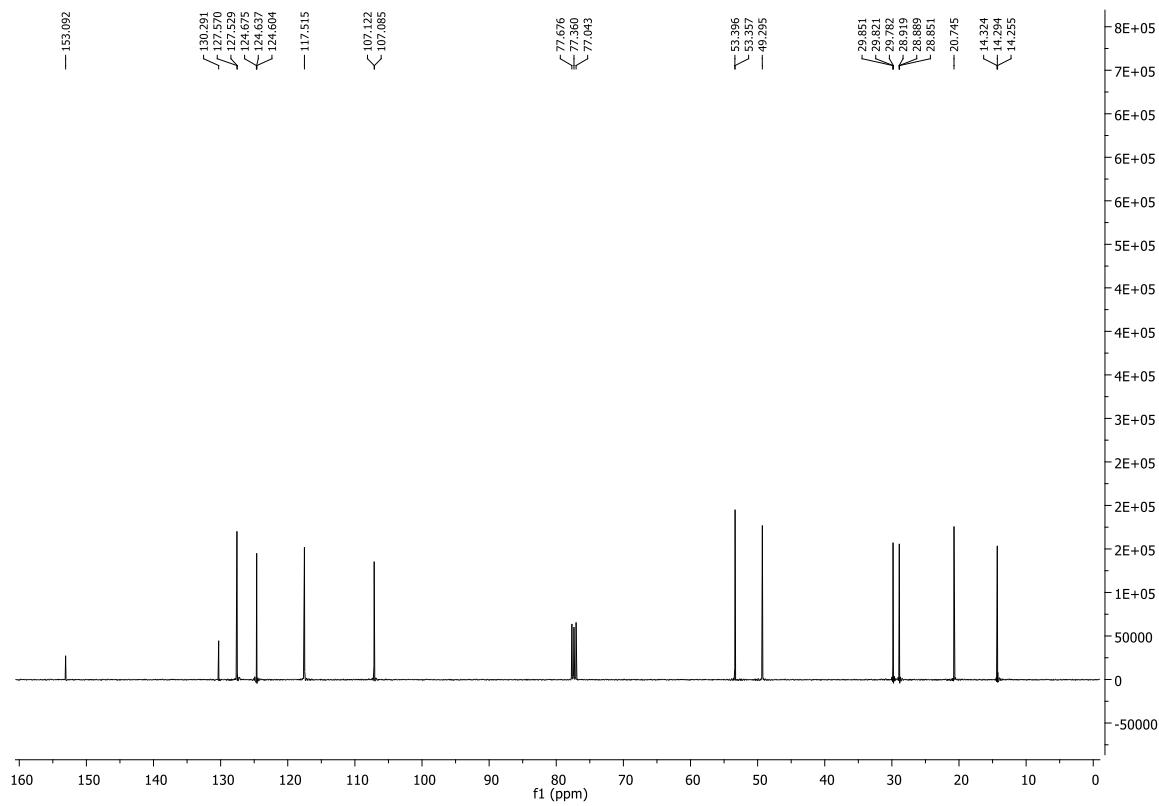
Substrate	R _t (min)
Compound 20: 2-Bromo-N-(4-methoxybenzyl)aniline	4.48
Iodobenzene (standard)	3.65

Compound 21: 1-Butylindoline

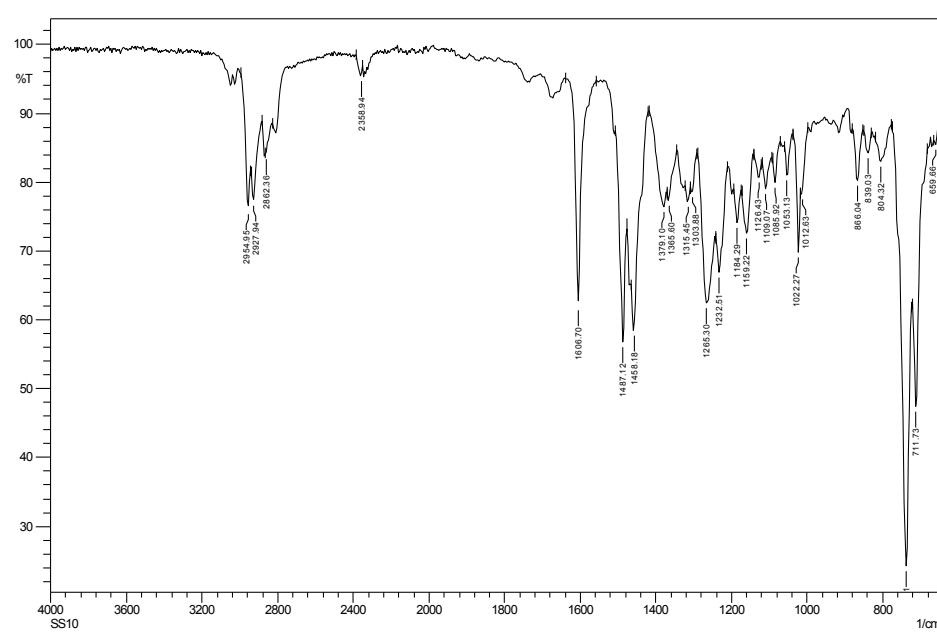
^1H NMR:



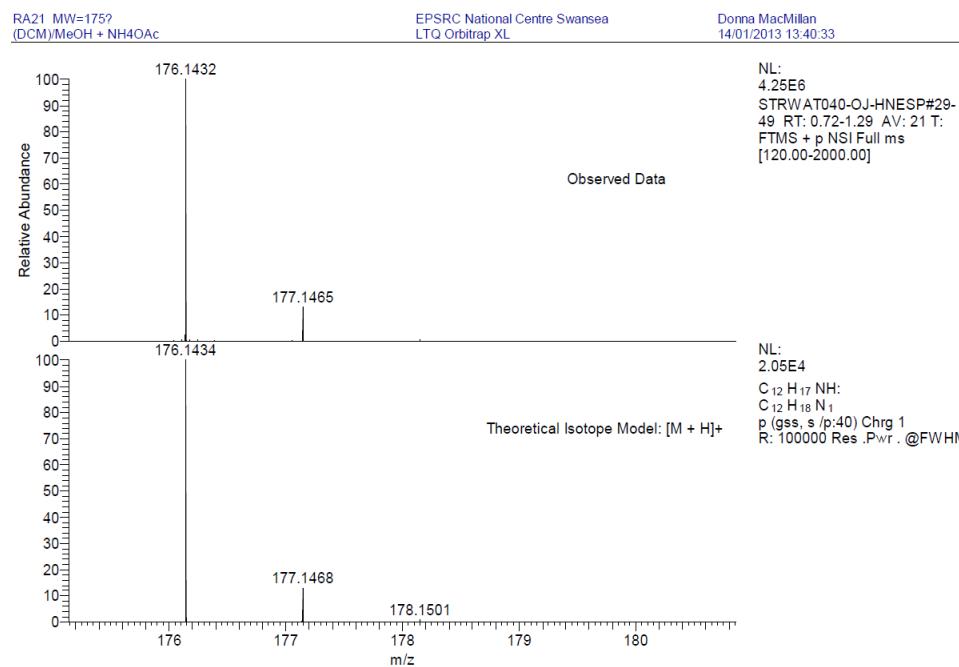
^{13}C NMR:



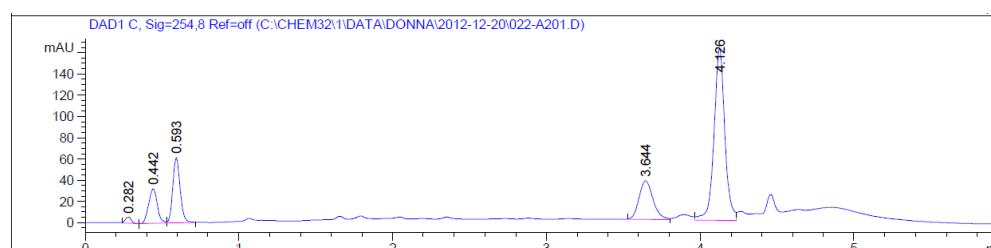
FTIR:



HRMS:



HPLC assay:



Substrate	R _t (min)
Compound 21: 1-Butylindoline	4.13
Iodobenzene (standard)	3.65