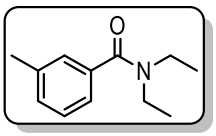
SUPPORTING MATERIAL

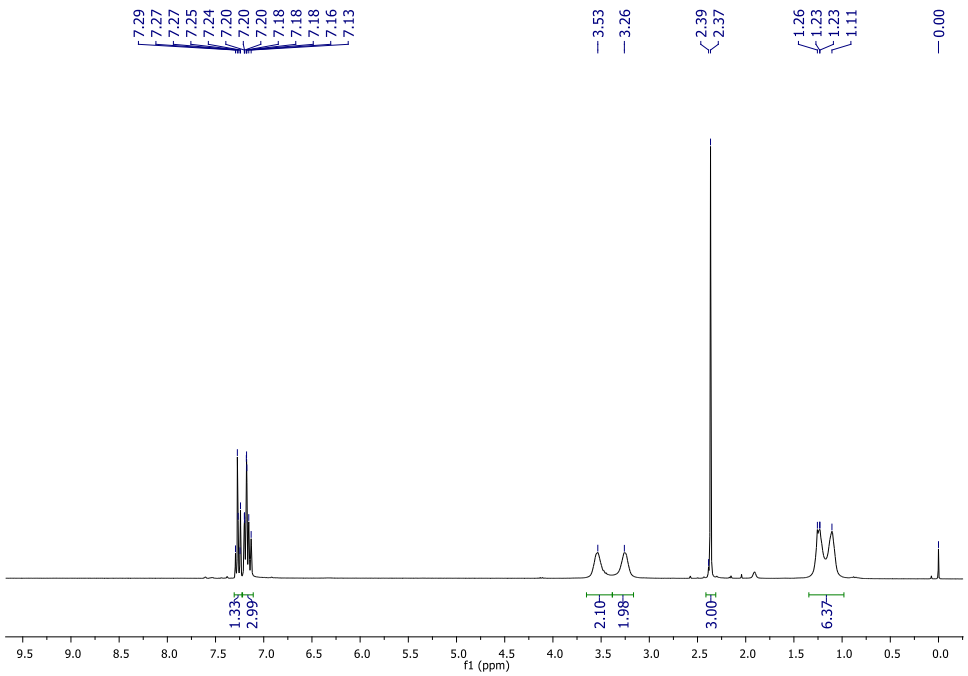
N,N-Diethyl-3-methylbenzamide

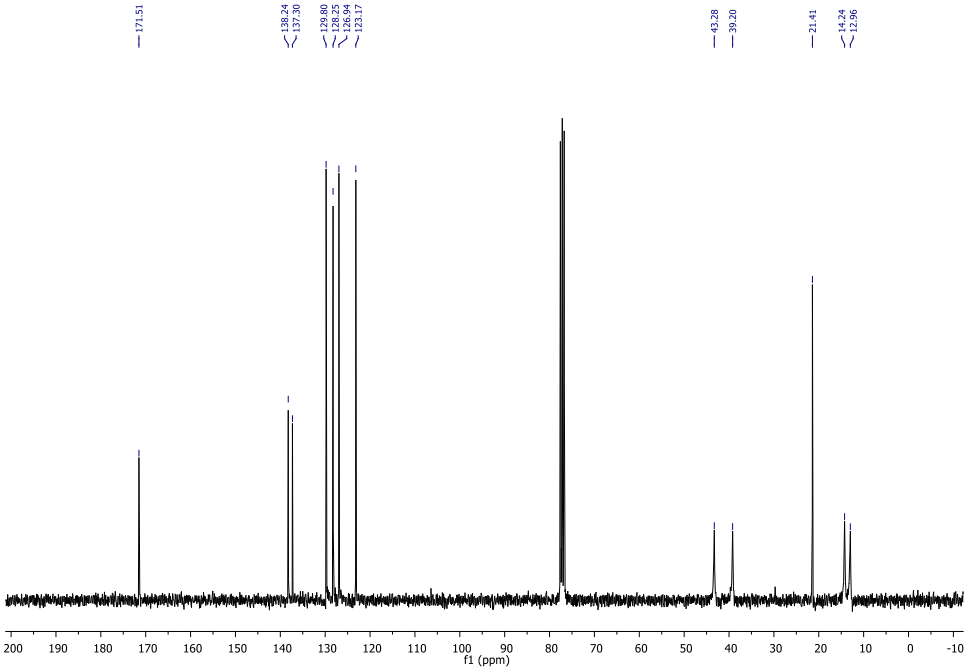
María Albert-Soriano and Isidro M. Pastor

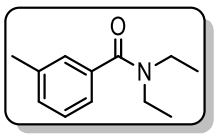
Organic Chemistry Department and Institute of Organic Synthesis (ISO), University of Alicante, ctra. San Vicente del Raspeig s/n, 03690, San Vicente del Raspeig, Alicante, Spain

N,*N*-Diethyl-3-methylbenzamide



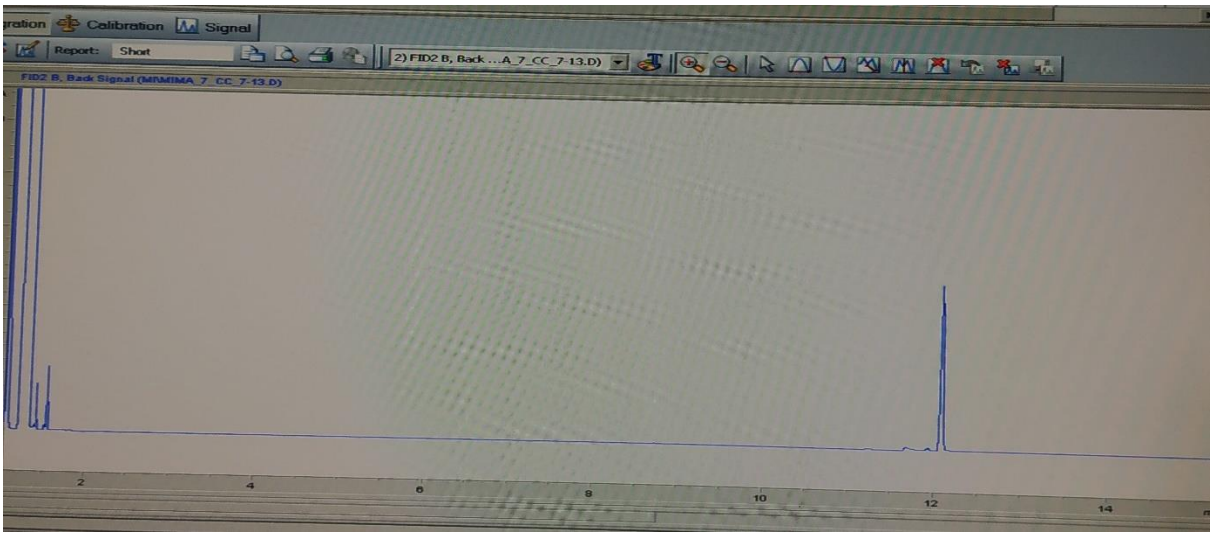




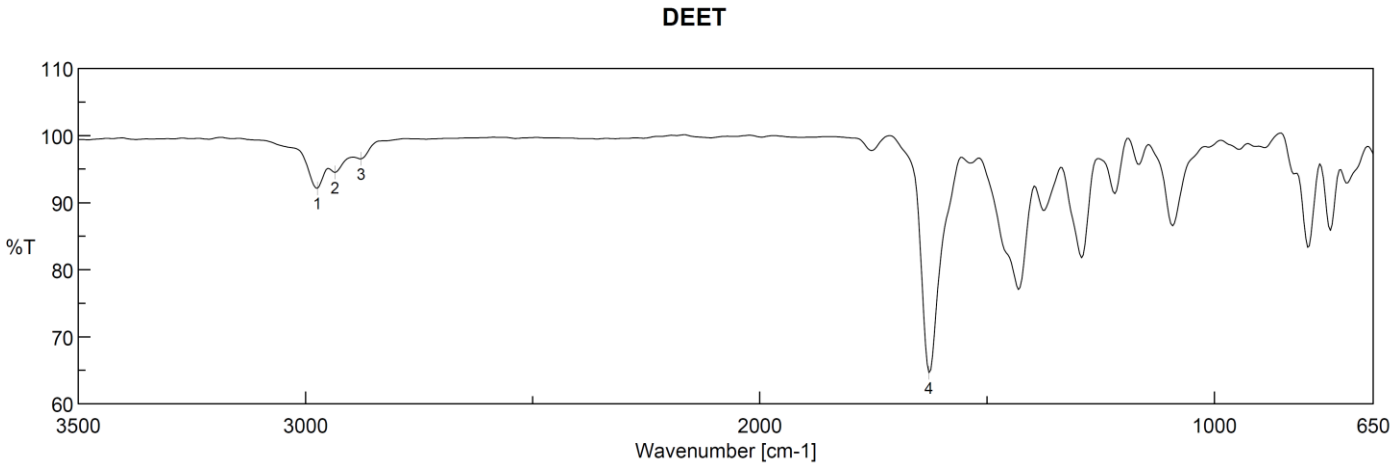


***N*,*N*-Diethyl-3-methylbenzamide**:[1] Pale yellow oil; purification by preparative TLC (Hexane/EtOAc 2:1), 66% yield; **IR** (ATR): *v* 2973, 2933, 2874, 1628 cm^{-1}; **^{1}H NMR** (300 MHz, $CDCl_3$): δ_H 7.31-7.23 (m, 1H, H_{Ar}), 7.22-7.11 (m, 3H, 3×H_{Ar}), 3.53, 3.26 (2 br s, 2H, 2H, 2×CH_2), 2.37 (s, 3H, $C_{Ar}CH_3$), 1.34-0.98 (m, 6H, 2×$CH_2C\mathit{H}_3$) ppm; **^{13}C NMR** (75 MHz, $CDCl_3$): δ_C 171.5 (CO), 138.2, 137.3 ($C_{Ar}CH_3$, $C_{Ar}CO$), 129.8, 128.2, 126.9, 123.2 (4×CH_{Ar}), 43.3, 39.2 (2×CH_2), 21.4 ($C_{Ar}CH_3$), 14.2, 13.0 (2×CH_2CH_3) ppm; **LRMS** (GC/MS-EI): *m/z* 191 [M^+] (21%), 190 (45), 119 (100), 91 (32), 65 (10).

GC – analysis

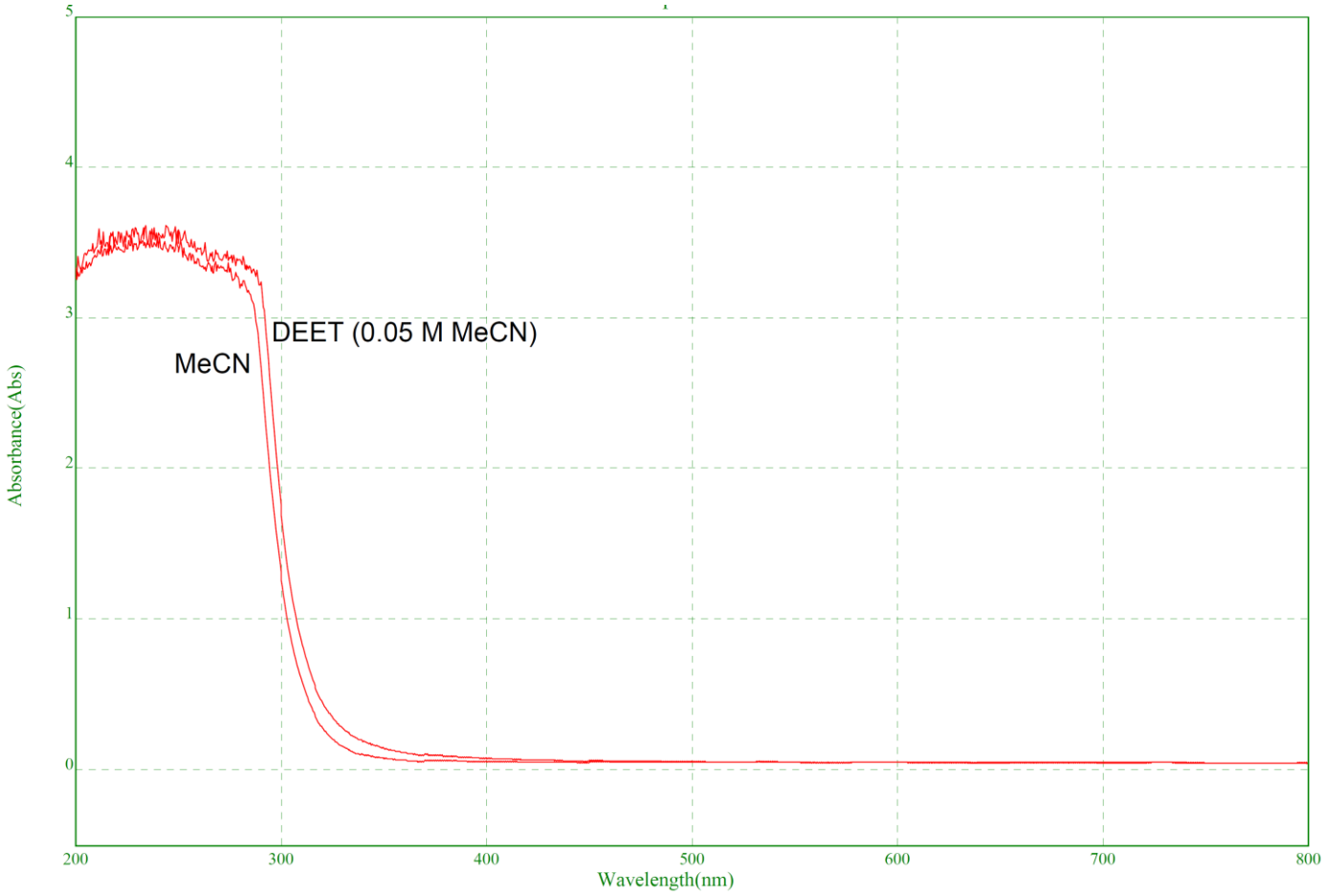


IR - analysis



[1] T. Shioiri, Y. Yokoyama, Y. Kasai and S. Yamada, *Tetrahedron* **1976**, *32*, 2211-2217.

UV-Vis spectrum



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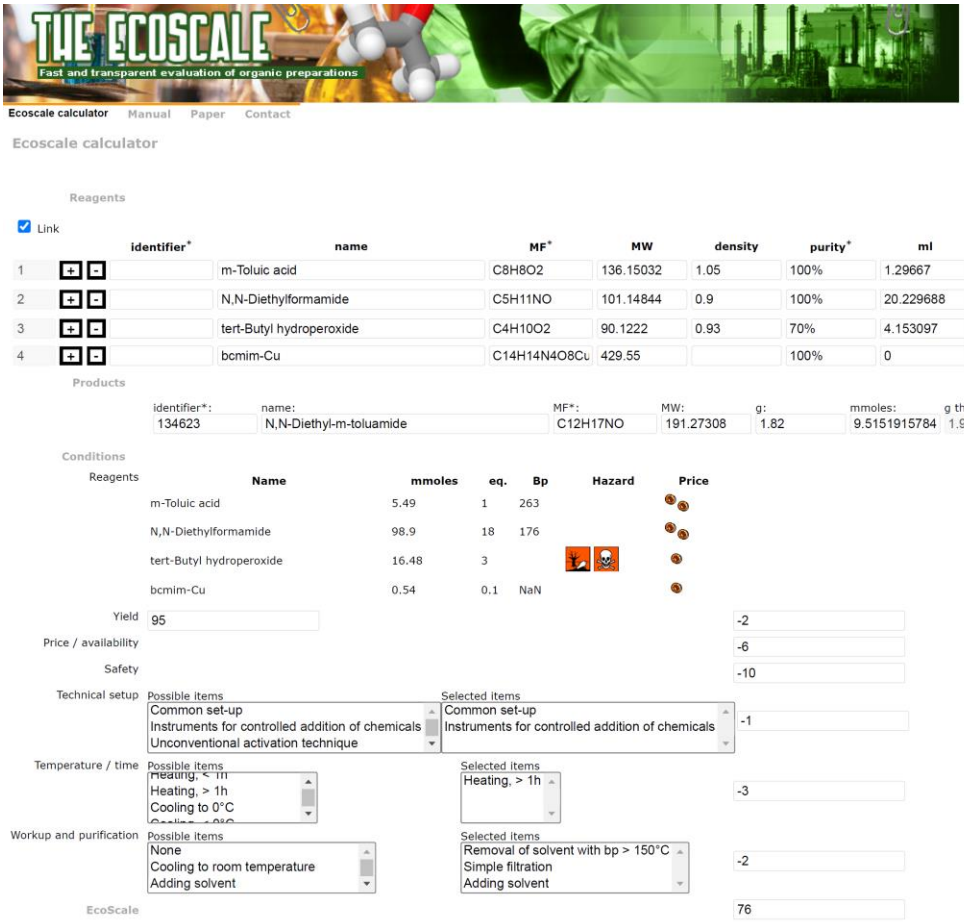
Scan Time: June 14 12:50:39 2022



EcoScale webpage:

http://ecoscale.cheminfo.org/calculator

Material Efficiency

PART ONE

Reactants	MW(g/mol)	Stoich. Coeff. (SC)	Adjusted MW (g/mol)	density (g/mL)	volume (mL)	mass used (g)		moles	comments		stoich. Moles	stoich. Mass (g)	mole ratio	mass ratio	excess mass (g)	mass kernel unreacted reagent (g)
3-methylbenzoic acid	136.15	1	136.15			1.36		0.0100		**limiting reagent**	0.01	1.36	1	1	0	0.06
DEF	101.15	1	101.15	0.908		18.21		0.1800			0.01	1.010385604	18.02282	18.022822	17.1996144	0.05
TBHP (70%)	90.12	1	90.12	0.93		2.7		0.0300			0.01	0.900207124	2.99931	2.9993097	1.799792876	0.04
		SUM	327.42			22.27	SUM					3.27		6.8092	19.00	0.16

Products	MW(g/mol)	Stoich. Coeff.	Adjusted MW (g/mol)
DEET	191.27	1	191.27
t-BuOH	74.12	1	74.12
H2O	18.02	1	18.02
CO2	44.01	1	44.01
		SUM	327.42

chemical equation is balanced

Reaction Solvents	MW(g/mol)	density (g/mL)	volume (mL)	mass used (g)		comments
				0	SUM	

Catalysts	MW(g/mol)	density (g/mL)	volume (mL)	mass used (g)		moles	comments
bcmim-Cu	430			0.43		0.00	
						#DIV/0!	
						#DIV/0!	
				0.43	SUM		

Workup Materials		density (g/mL)	volume (mL)	mass used (g)		comments
				0	SUM	

Purification Materials	[redacted]	density (g/mL)	volume (mL)	mass used (g)		comments
Pad of silica				2		
Hexane		0.655	10	6.55		
EtOAc		0.902	10	9.02		
				17.57	SUM	

TOTAL INPUT MASS				40.27	g

Target Product	MW(g/mol)	moles collected		mass collected	
DEET	191.27	0.00952	**MASS OF TARGET PRODUCT**	1.82	g

MASS RAW WASTE = TOTAL INPUT MASS - MASS OF TARGET PRODUCT			38.45	g

PART TWO

Recycling-Recovery of Materials

Item	[redacted]	mass recovered	
DEF		16	g
bcmim-Cu		0.43	g
			g
	MASS RECOVERED MATERIALS	16.43	SUM

MASS ADJUSTED WASTE = MASS RAW WASTE - MASS RECOVERED MATERIALS	22.02	g

E-factor (E) = mass adjusted waste / mass product	12.099	

Process Mass Intensity (PMI) = (total input mass - mass recovered materials) / mass product	13.099	

Reaction Mass Efficiency (RME) = 100 * (mass product / (total input mass - mass recovered materials))	7.6	%

Atom Economy (AE) = 100 * (MW product / sum of MWs of reactants)	58.4	%

Yield (Y) = 100 * (moles product / moles limiting reagent) * (SC limiting reagent / SC Product)	95.3	%

CHECK CALCULATIONS

PMI = 1 + E	13.099

RME = 1/(1 + E)	0.1

PART THREE

Metrics Report Summary

Actual Mass of Reagents	22.27	g	
Stoichiometric Mass of Reagents	3.27	g	
Stoichiometric Factor	6.809163307		
Materials Recovery Parameter	0.934144295		
Raw E-factor profile			
E-kernel	0.797028972		
E-excess	10.43923476		
E-rxn solvent	0		
E-catalyst	0.236263736		
E-work-up	0		
E-purification	9.653846154		
E-aux	9.89		
E-total	21.12637363		
Yield	95.3	%	
AE	58.4	%	
PMI	13.099		
RME (global)	7.6	%	

Parameter	**Actual**	**Ideal Limit**
AE	0.584	1
Rxn Yield	0.953	1
1/SF	0.147	1
MRP	0.934144295	1
RME	0.076	1
VMR	0.655545028	
(vector magnitude ratio)		