Supplementary Information (SI) for Reaction Chemistry & Engineering.
This journal is © The Royal Society of Chemistry 2025
Please do not adjust margins

ARTICLE

Supplementary Material

The energetical analysis of a method for the scale-up of a microwave flow P-esterification by recirculation

József Schindler[a]*, István Greiner[b] and György Keglevich[a]

[a] *Department of Organic Chemistry and Technology, Faculty of Chemical Technology and Biotechnology, Budapest University of Technology and Economics, 1111 Budapest, Műegyetem rkp. 3, Hungary. E-mail:*
** schindler.jozsef@vbk.bme.hu†*
[b] *Gedeon Richter Plc,*

General Information.

The MW reactions were carried out in a CEM Discover (300 W) focused microwave reactor (CEM Microwave Technology Ltd., Buckingham, U.K.) equipped with a 10 mL flow cell available from the supplier of CEM using 40–200 W irradiation under isothermal conditions. The reaction temperature was monitored by an external IR sensor. LC–MS measurements were performed with an Agilent 1200 liquid chromatography system, coupled with a 6130 quadrupole mass spectrometer equipped with an ESI ion source (Agilent Technologies, Palo Alto, CA, USA).

Preparation of the reaction mixture

A mixture of 10 g (70.4 mmol) $Ph_2P(O)H(OH)$ and 100 mL (1.1 mol) of butyl alcohol was homogenized by stirring at 25°C for 15 min. For the catalyzed versions, 1.0 g (3.5 mmol) of [bmim][PF_6] was also added to another mixture prepared as above.

General procedure for the circulated flow esterification of phenyl-H-phosphinic acid (1) with butyl alcohol

A 25, 50 or 100 mL mixture containing PhP(O)H(OH) (**1**) and nBuOH in a ratio of 1: 15.6, in the catalyzed versions also 5% of [bmim][PF6] was circulated applying the corresponding rate at 25°C and under 17 bar. After the mixture filled the system, the flow cell was irradiated with a power of 300 W until the desired temperature was reached. After this, the power was controlled automatically (by 40–200 W) to maintain the value set. The operation was regarded steady state after 17–175 min reaching the “plato” as suggested by LC-MS analysis.
A part of the measurements was repeated, and the data could be reproduced within a standard deviation ± 2%.

Table S1 Direct esterification of phenyl-*H*-phosphinic acid (1) in a recirculated batch MW a reactor

Entry	W [mL/min]	T [°C]	Cat.	V [mL]	$t_{0.9Xf}$ [min]	$0.9X_f$ [%]	$n_{t0.9Xf}$ [mmol]	P_f [W]	Energy [Wh]				Molar energy [Wh/mmol]			Avarage power [W]			Y [mmol/min]
									E_t	E_b	E_r	EE	E_{mt}	E_{mb}	E_{mr}	$\bar{P}_t$	$\bar{P}_b$	$\bar{P}_r$	
1	1	160	-	25	77.0	62.6	11.0	42	62	54	8	0.14	5.6	4.9	0.7	48	42	6	0.14
2	1	160	yes	25	31.0	62.5	11.0	24	22	12	9	0.76	2.0	1.1	0.9	42	24	18	0.35
3	1	140	yes	25	41.7	59.4	10.4	19	27	13	14	1.04	2.6	1.3	1.3	39	19	20	0.25
4	3.5	160	-	25	13.6	68.3	12.0	108	64	24	39	1.60	5.3	2.0	3.3	281	108	173	0.88
5	3.5	160	yes	25	10.7	68.6	12.1	105	47	19	29	1.53	3.9	1.6	2.4	265	105	160	1.13
6	6.5	160	-	25	14.3	68.0	12.0	148	71	35	36	1.01	5.9	2.9	3.0	297	148	149	0.84
7	6.5	160	yes	25	10.7	72.1	12.7	145	53	26	27	1.04	4.2	2.0	2.1	296	145	151	1.18
8	10	160	-	25	15.2	72.1	12.7	205	75	52	23	0.45	5.9	4.1	1.9	298	205	93	0.83
9	10	160	yes	25	10.7	72.3	13.4	202	56	37	19	0.52	4.2	2.7	1.5	296	202	94	1.17
10	10	160	-	50	23.5	67.1	23.6	185	115	72	43	0.59	4.9	3.1	1.8	294	185	109	1.00
11	10	160	-	100	73.0	63.5	44.7	180	273	219	54	0.25	6.1	4.9	1.2	224	180	44	0.61