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Microwave irradiation in organic synthesis

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Abstract. Studies on applications of microwave irradiation to organic synthesis published over 2000–2002 are surveyed. The theoretical concepts concerning the influence exerted by microwave irradiation on the course of organic reactions are briefly discussed. The bibliography includes 354 references.

I. Introduction

Nowadays, great investigation efforts in diverse fields of science and industry are put into the development of resource- and energy-conserving environmentally friendly technologies. In this regard, microwave irradiation (hereinafter, MWI) is becoming increasingly important, since it enables exceptional intensification of various processes, including those that cannot be optimised using traditional techniques.

The pioneering work on the utilisation of MWI as an efficient heating source dates back to the late 1940s, and over the 1970s and 1980s it came into common use in inorganic and analytical chemistry for analysis of various objects, drying and dehydration of samples, synthesis of inorganic materials, elucidation of polymorph transitions, *etc*.[1] An impetus to the use of MWI in organic synthesis was given by the studies of Gedye *et al*.[2] and Giguere *et al*.,[3] who observed a significant reduction in the time required for the completion of several classical chemical reactions under MWI irradiation as compared to traditional heating conditions. Other advantages of MWI included the saving of electric power or other energy resources, the possibility of conducting a reaction at an elevated pressure at a temperature significantly exceeding the boiling point of the solvent used or under solvent-free conditions, the synthesis of compounds that are unattainable or difficult to prepare with other activation methods.

These factors favouring the use of MWI for the activation of organic reactions were rapidly appreciated by synthetic chemists. It is of note that the wide acceptance of the method was partly due to the simplicity of the experiments: they only required availability of an ordinary commercial MW-oven. Nevertheless, the use of such ovens for the realisation of microwave-assisted chemical reactions have several drawbacks:

— the ovens are not specifically suited for chemical experiments;

— the microwave field in a reaction vessel is inhomogeneous;

— changes in temperature and pressure of a reaction mixture during an experiment cannot be controlled;

— the MWI power varies over time.

All these factors result in poor reproducibility of results and sometimes give rise to explosion or ignition of solvents. Dedicated chemical microwave reactors are now available commercially, which provide homogeneous distribution of the electromagnetic field throughout the reaction volume and computer-controlled adjustment of the radiation power. Such reactors are normally equipped with magnetic stirrers and temperature and pressure gauges. Such special reactors are one order of magnitude more expensive than normal household MW-ovens and thus ~70% of experiments are still performed using the latter.

Several aspects of the application of MWI to organic synthesis have been surveyed in previous reviews, including the theoretical background and recent achievements of MW-assisted organic synthesis,[4–8] solvent-free synthesis,[9] reactions on solid sup-

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ports,[10,11] synthesis under the conditions of phase-transfer catalysis,[12] preparation of heterocyclic compounds [13] and cycloaddition reactions.[14]

Despite the great number of experimental works describing practical aspects of MWI utilisation, the theory of microwave-assisted organic synthesis has not been elaborated in sufficient detail. Potentially, MWI affects the course of a chemical process in several ways, including purely thermal effects (heating rate, overheating or 'hot spots'), the selective absorption of the radiation by polar compounds and the specific microwave effect (MWE) related to the nonthermal action of MWI.

The term MWE first introduced by Perreux and Loupy [6] is now frequently quoted in studies related to microwave-assisted organic synthesis. Based on the well-known Hammond postulate,[15] Perreux and Loupy suggested [6] that the transition state is close to the ground state for reactions characterised by low activation energies. In such cases, the difference in polarities of the transition and ground states is small and thus the microwave effect is insignificant. In contrast, for reactions characterised by high activation energies, the transition state is rather close to the reaction products. This means that the polarity change in the course of the reaction is prominent and thus the MWE is enhanced.

If polar aprotic solvents strongly absorbing MWI are used, it is the solvent molecules that predominantly feed the reagents with energy (*i.e.*, the energy transfer is indirect) and therefore the MWE is expected to be masked with MWI absorption by the solvent. As a result, the difference in the reaction rates under the conditions of microwave irradiation and convection heating is diminished.

On the other hand, if nonpolar solvents (*e.g.*, CCl_4, alkanes, benzene, *etc.*) weakly interacting with microwaves are used, the MWI directly interacts with the reagents, and the MWE in its pure form is manifested.[6]

The MWE is most prominent under solvent-free conditions, *i.e.*, when no external factors hinder the interaction of microwaves with reagents such that the overall MWI absorption is determined solely by their nature.

Unfortunately, in the vast majority of studies devoted to microwave organic synthesis, the influence of the MWI power, the stirring intensity and the reaction medium type on the reaction rates and yields of products were not considered systematically. Thus, the aim of the present review is to serve as a general source of reference data. It covers microwave-assisted syntheses of various classes of organic compounds published over the period from 2000 to 2002. Earlier studies have been reviewed by Lindstrom *et al.*[7] and are not cited here.

The literature data presented below are grouped according to the types of organic compounds concerned and reactions involving them.

II. Formation of new C = C and C – C bonds under microwave irradiation

The most common reactions utilised for the formation of carbon – carbon bonds are the aldol-crotonic condensation or related reactions (examples Nos 1 – 7 in Table 1); the condensation of nitromethane or its derivatives with 5-nitro-6-chloromethylimidazothiazole (No. 8), benzaldehyde or its derivatives (No. 9); Wittig reaction (Nos 10 – 12) and elimination reactions (No. 13).

Unfortunately, comparative data on different activation methods were reported only in a few studies.[22,25,26] In particular, Gellis *et al.*[22] showed that the microwave-assisted condensation of

Table 1. Microwave-assisted formation of new C = C and C – C bonds.

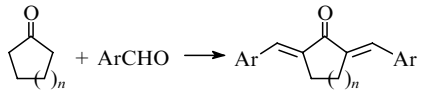
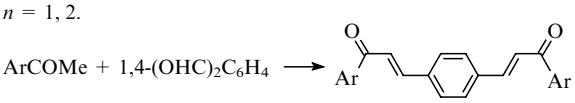
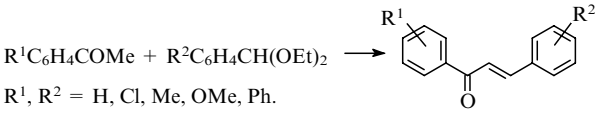
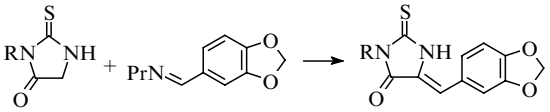
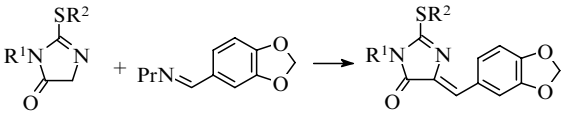
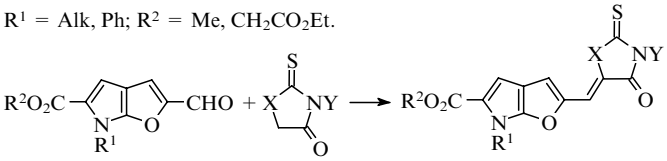
No.	Reaction	Conditions	Number of examples	Yield (%)	Ref.
Formation of new C = C bonds					
1	 n = 1, 2.	KF/Al_2O_3, MW, 2 – 5 min	9	75 – 90	16
2		NaOH – EtOH, MW, 2 – 4 min	7	66 – 85	17
3	 R^1, R^2 = H, Cl, Me, OMe, Ph.	Lewis acids, MW, 8 min	11	0 – 84	18
4	 R = Alk, Ph.	MW, 1 h	3	75 – 87	19
5	 R^1 = Alk, Ph; R^2 = Me, CH_2CO_2Et.	MW, 1 h	6	71 – 92	19
6	 R^1, R^2 = H, Alk; X = NH, S; Y = H, NH_2.	AcONa, MW, 2 – 8 min	7	50 – 89	20

Table 1 (continued).

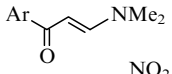
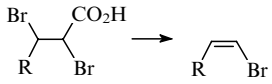
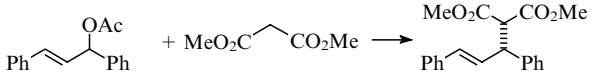
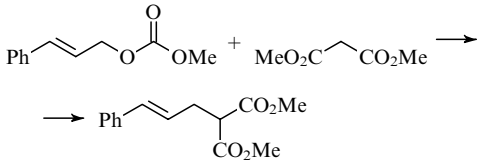
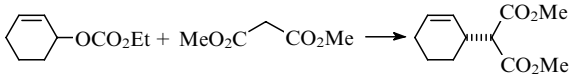
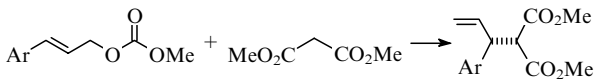
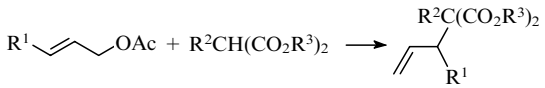
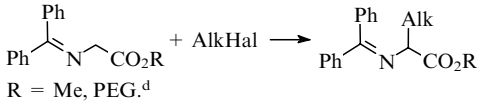
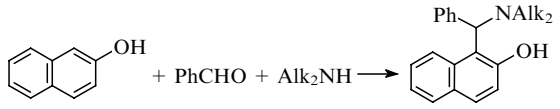
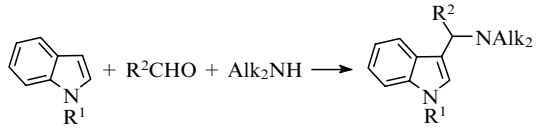
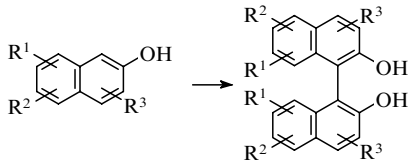
No.	Reaction	Conditions	Number of examples	Yield (%)	Ref.
	Formation of new C=C bonds				
7	$ArCOMe + Me_2NCH(OMe)_2 \longrightarrow$ 	MW, 3–9 min	8	83–95	21
8	 R^1, R^2 = H, Alk, Het; M = Li, NBu_4.	*a*) H_2O, Li^+, MW, 6 min; *b*) Bu_4N^+, Δ, 24 h	9 9	48–98 45–99	22 22
9	$ArCHO + MeNO_2 \longrightarrow ArCH{=}CHNO_2$	K_2CO_3/Al_2O_3, MW, 4–6 min	12	71–95	23
10	$R^1CH_2Br + R^2CHO +$  $\longrightarrow R^1CH{=}CHR^2$ R^1 = CN, CO_2Me, Ph; R^2 = Ar, Het.	K_2CO_3, MeOH, MW, 5 min	15	11–95	24
11	$Ph_3P{=}CR^1R^2 + ArCHO \longrightarrow ArCH{=}CR^1R^2$ R^1 = COMe, CO_2Et, CN, CON(Me)OMe; R^2 = H, Me.	*a*) DMSO, MW, 5 min *b*) THF, Δ, 1 h	25 10	48–100 16–92	25 25
12	$Ph_3P{=}C(I^+Ph)(CO_2Et)\ BF_4^- + RCHO + Bu_4NHal \longrightarrow$ $\longrightarrow RCH{=}C(CO_2Et)Hal$ $R = Pr^n$, Ar.	*a*) DMF ($CHCl_3$, MeCN, *etc.*), MW, 2–5 min *b*) Δ, 8–32 min	13 13	68–97 61–97	26 26
13	 R = Alk, Ar.	Et_3N, DMF, MW, 0.2–1 min	15	73–99	27
	Formation of new C–C bonds				
14		$[(\eta^3\text{-}C_3H_5)PdCl]_2$, AcOK, BSA,[a] L[b] *a*) MW, 0.25–15 min *b*) Δ, 4.5–5760 min	 17 6	 85–99 56–99	28
15		$[(\eta^3\text{-}C_3H_5)PdCl]_2$, BSA,[a] THF, MW, 5 min	1	73	29
16		$[(\eta^3\text{-}C_3H_5)PdCl]_2$, L,[b] MW, 1.5–6 min	5	65–83	28
17	 Ar = Ph, *p*-RC_6H_4.	*a*) Ar = Ph: $Mo(CO)_6$, L,[b] PPh_3, BSA,[a] THF, MW, 4–9 min *b*) Ar = Ph, *p*-RC_6H_4: $Mo(CO)_6$, L,[b] $NaCH(CO_2Me)_2$, BSA,[a] THF, MW, 5–6 min	10 8	59–84 50–89	29 30
18	 R^1 = H, Ph; R^2 = H, Me; R^3 = Me, Et.	$Pd_2(dba)_3$ (see[c]), KF/Al_2O_3, MW, 5–30 min	4	30–98	31
19	 R = Me, PEG.[d]	PEG,[d] M_2CO_3 (M = K, Cs), MW, 15–60 min	8	75–98	32

Table 1 (continued).

No.	Reaction	Conditions	Number of examples	Yield (%)	Ref.
Formation of new C – C bonds					
20		Al_2O_3, MW, 5 min	3	68 – 73	33
21	 R^1 = H, Alk, R^2 = H, *p*-$NO_2C_6H_4$.	Al_2O_3, MW, 4 – 5 min	4	50 – 91	33
22	 R^1 = H, Me, CO_2Me; R^2 = H, Br; R^3 = H, OH, OMe.	$FeCl_3 \cdot 6H_2O$, MW, 4 – 20 min	8	80 – 97	34

[a] BSA is *N*,*O*-bis(trimethylsilyl)acetamide; [b] L means different diamide ligands; [c] dba is dibenzylideneacetone; [d] PEG is polyethylene glycol.

chloromethylimidazothiazole with 9 nitromethane derivatives proceeded 240 times faster than the respective reactions under thermal activation (see Table 1). Evidently the MWE can be observed here.

The microwave-assisted introduction of double bonds into organic molecules following the Wittig method (reactions Nos 10 – 12) is slightly accelerated as compared to the thermally activated reaction; the use of reagents grafted to a solid substrate does not result in a further reduction in the reaction time.

Table 1 also lists some examples of the formation of new single C – C bonds under MW conditions, including allyl substitution, C-alkylation, C-aminoalkylation and oxidative coupling reactions (Nos 14 – 22).† Unfortunately, no conclusions on the MWE involvement can be deduced in those cases owing to the absence of comparative data. Among them, the work by Kaiser *et al.*[29] is worth a special mention, where the allyl alkylation of methyl-(*E*)-3-phenylallylcarbonate (No. 17) was studied and the influence of several factors, such as the nature of catalyst (*cf.* with No. 15) and solvent, radiation power and grafting of the reagent on a solid substrate, on the yield of the target product was analysed.

III. Reactions involving multiple carbon – carbon bonds

Table 2 lists examples of reactions involving C = C bonds, which are activated by microwave irradiation. The hydrogenation of double bonds is illustrated with three examples (Nos 1 – 3). These data show that the microwave-assisted hydrogenation proceeds nearly as fast as the thermally activated reaction but the use of a support in the former case results in a 360-fold acceleration.

The C-arylation of compounds containing a double bond in the presence of Pd-based catalysts is represented by reactions Nos 4 – 7. Olofsson *et al.*[40] found that the microwave-activated C-arylation following the Heck method proceeds 200 times faster than the thermally activated one (reaction No. 6, Table 2).

In the case of the addition of aldehydes to alkenes with non-activated double bonds (reaction No. 8), the yields of target products are virtually the same for the microwave and thermal activations but the use of MWI gives rise to a 24-fold acceleration of the reaction.

The addition of compounds with reactive methylene or methine groups to alkenes with activated double bonds (*i.e.*, the Michael addition) is illustrated with reactions Nos 9 – 14 (see Table 2).

Michaud *et al.*[47] showed that the double Michael addition of benzylidenecyanoacetic esters to nitromethane (reaction No. 13) catalysed by piperidine followed by intermolecular cyclisation yields a mixture of functionalised cyclohexenes with either two (compound **A**) or one (compound **B**) methoxycarbonyl groups in a ratio dependent on the MWI parameters and amount of piperidine. For instance, with piperidine taken in equimolar proportion to nitromethane, the monomethoxycarbonyl product (**B**) is predominantly formed, and *vice versa,* the maximum yield (80%) of dimethoxycarbonylcyclohexene (**A**) is achieved with piperidine used in catalytic amounts.

The microwave-assisted Diels – Alder cycloaddition reactions that yield polycyclic systems with isolated double bonds are significantly accelerated both in the presence (reaction No. 16, see Table 2) and in the absence (reaction No. 15) of solvents as compared to the respective thermally activated reactions. It has to be noted that, although the yields of products under different conditions are comparable, some reactions (*e.g.*, see Ref. 50) can be realised only under microwave irradiation.

Table 3 gives some examples of catalytic reactions involving acetylene derivatives, such as Mannich aminomethylation (reactions Nos 1, 2), Sonogashira cross-coupling (No. 3 – 6), desilylation (No. 7), Glaser synthesis of conjugated diacetylene derivatives (No. 8), metathesis (No. 9) and C-acylation (No. 10).

The data presented indicate that the reaction times in the case of MWI activation are substantially reduced as compared to the

† For other examples of the formation of C – C bonds, see Table 2, which lists reactions involving C = C bonds.

Table 2. Reactions involving the C=C bond.

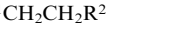
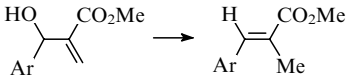
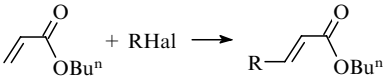
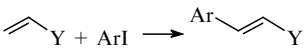
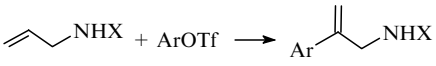
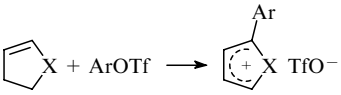
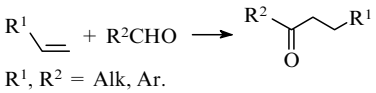
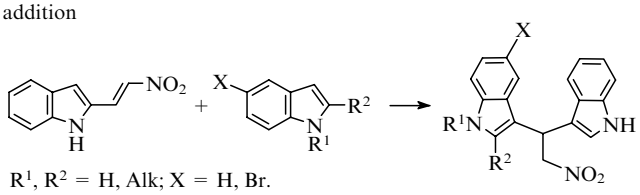
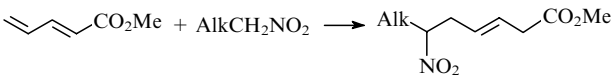
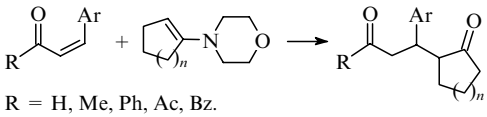
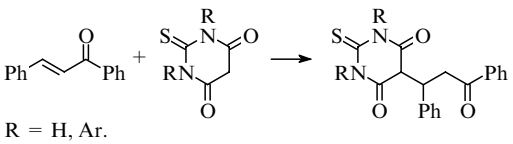
No.	Reaction	Conditions	Number of examples	Yield (%)	Ref.
Hydrogenation					
1	$R^1CH{=}CHR^2 \longrightarrow R^1CH_2CH_2R^2$ R^1 = Et, Ph; R^2 = CO_2Alk, C(O)Me, C(O)NMe_2, CN, CHO.	*a*)  RhCl(PPh)$_3$, DMSO, MW, 30 s	8	80–95	35
		b) Δ, 3 h	8	50–95	35
2	PhCH=CHR ⟶ $PhCH_2CH_2R$ R = Ph, CO_2Me.	10% Pd/C, HCO_2NH_4 or HCO_2NEt_4, MW, 80 min	4	83–99	36
3		$NaBH_4/Al_2O_3$, MW, 8–12 min	6	72–90	37
C-Arylation					
4	 R = Ar, Het.	$PdCl_3$, P(Tol-*o*)$_3$, (bmim)PF_6 (see [a]), Et_3N, MW, 5–45 min	7	39–95	38
5	 Y = Ph, CO_2H, CO_2Me.	$Pd(PPh_3)_2Cl_2$, Bu^n_4NBr, K_2CO_3, H_2O, MW, 10 min	7	86–93	39
6	 X = Boc, Pht; Ar = Ph, 1-Naph, *p*-$MeOC_6H_4$, *etc*.	$Pd(OAc)_2$, $(C_5H_4PPh_2)_2Fe$, K_2CO_3, MeCN			40
		a) MW, 3–6 min	14	6–68	
		b) Δ, 20 h	14	7–74	
7	 X = CH_2, O.	$[Pd_2(dba)_3]$L, benzene, MW, 10–480 min	11	53–85 [b]	41
Addition to a non-activated double bond					
8	 R^1, R^2 = Alk, Ar.	*a*) 2-NH_2-3-MeC_5H_3N, MW, 10 min	11	0–95	42
		b) PhMe, Δ, 4 h	11	13–93	42
Michael addition					
9	 R^1, R^2 = H, Alk; X = H, Br.	*a*) SiO_2, MW, 7–10 min	6	70–86	43
		b) room temperature, 8–14 h	6	69–84	43
10		Amberlyst A, MW, 7 min	8	55–75	44
11	 R = H, Me, Ph, Ac, Bz.	Al_2O_3, MW, 5–8 min	13	80–89	45
12	 R = H, Ar.	Al_2O_3, MW, 1.5–2.3 min	6	83–88	46

Table 2 (continued).

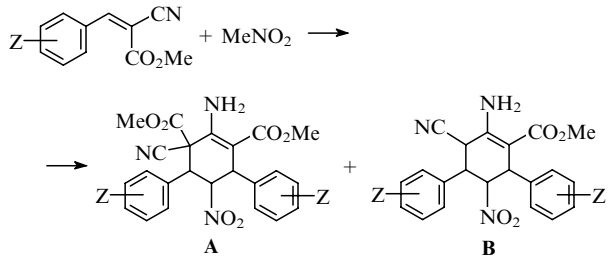
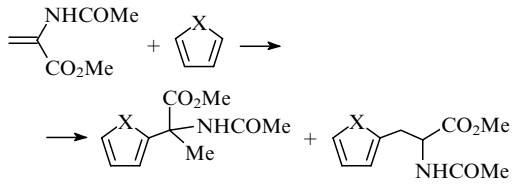
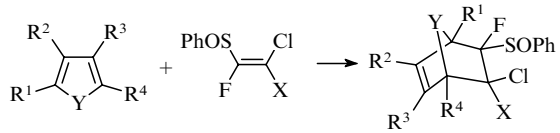
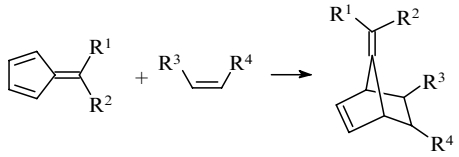
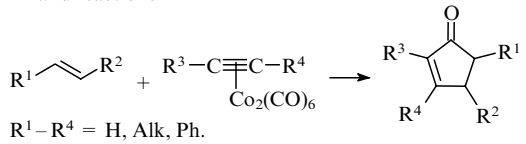
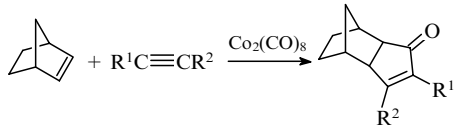
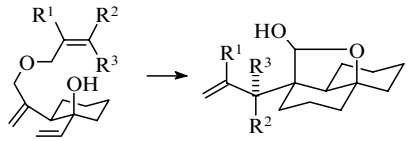
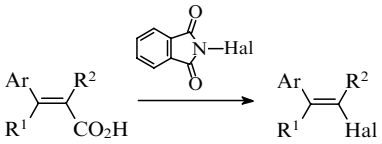
No.	Reaction	Conditions	Number of examples	Yield (%)	Ref.
Michael addition					
13	 Z = H, Hal, OMe, NO_2.	piperidine, MW, 11 – 25 min	7	0 – 80 (**A**) 100 – 20 (**B**)	47
14	 X = O, NH, NBn.	Si(M) [c] (M = Zn, Al, Ti), MW, 15 – 30 min	10	> 3 – 70 [b]	48
Diels – Alder reactions					
15	 R^1, R^4 = H, Ph; R^2, R^3 = H, Alk; Y = CH_2, O; X = F, Cl.	*a*) MW, 3 – 25 min *b*) PhMe, Δ, 10 – 24 h	7 5	71 – 90 70 – 90	49 49
16	 R^1, R^2 = H, Alk; R^3, R^4 = CO_2Me, C(O)OC(O), C(O)NHC(O), C(O)CH=CHC(O).	*a*) DMSO, MW, 2 h *b*) DMSO, Δ, 5 h *c*) benzene, Δ, 5 h	9 6 9	30 – 78 0 – 75 0 – 89	50 50 50
Pouson – Khand reactions					
17	 R^1 – R^4 = H, Alk, Ph.	$C_2H_4Cl_2$, MW, 2 – 20 min	9	11 – 90	51
18	 R^1 = Alk, Ph, CO_2Me; R^2 = H, CO_2Me.	cyclo-$C_6H_{11}NH_2$, MW, 5 min	4	48 – 81	52
Cascade cyclisation reactions					
19		PhMe, DBO,[d] MW, 60 min	7	60 – 98	53

Table 2 (continued).

No.	Reaction	Conditions	Number of examples	Yield (%)	Ref.
Substitution reactions					
20	 R^1 = H, Me, Ph; R^2 = H, Me.	LiOAc, MeCN – H_2O, MW, 1 – 2 min	15	33 – 96	54

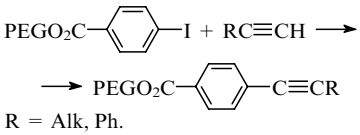
[a] bmim is 1-butyl-3-methylimidazole; [b] the total yield of isomers is given; [c] silicon-supported metals; [d] DBO is 1,4-diazabicyclo[2.2.2]nonane.

thermal activation. In particular, the aminomethylation is accelerated by a factor of 360 (reaction No. 2, see Table 3). Meanwhile, MWI gives no improvement in terms of yields of reaction products, and they even decrease in a few cases (*e.g.*, reaction No. 6).

IV. Synthesis of aromatic compounds and reactions with them

Table 4 lists selected examples of the use of MWI in the synthesis (reactions Nos 1 – 4) and modification of aromatics, *viz.*, Suzuki

Table 3. Microwave-assisted reactions involving the C ≡ C bond.

No.	Reaction	Conditions	Number of examples	Yield (%)	Ref.
1	$R^1C{\equiv}CH + (CH_2O)_n + R^2R^3NH \longrightarrow R^1C{\equiv}CCH_2NR^2R^3$ R^1 = Alk, Ar; R^2 = Alk, Ph; R^3 = Alk.	CuI/Al_2O_3, MW, 4 min	8	40 – 90	55
2	$R^1C{\equiv}CH + HCHO + R^2R^3NH \longrightarrow R^1C{\equiv}CCH_2NR^2R^3$ R^1, R^2 = Alk, Ph; R^3 = Alk.	*a*) $CuCl/Al_2O_3$, MW, 1 min *b*) Δ, 6 h	13 13	70 – 94 73 – 95	55 55
3	$R^1C{\equiv}CH + R^2I \longrightarrow R^1C{\equiv}CR^2$ R^1 = CH_2OH, Ph, $C_6H_4NO_2$-*p*; R^2 = Alk, Ar.	$CuI - PPh_3$, K_2CO_3, DMF, argon *a*) MW, 10 min *b*) Δ, 10 – 24 h	 7 7	 80 – 92 75 – 95	56
4	 R = Alk, Ph.	$Pd(PPh_3)_2Cl_2$, CuI, K_2CO_3, MW, 1.5 min	6	68 – 91	57
5	$R^1C{\equiv}CX + R^2I \longrightarrow R^1C{\equiv}CR^2$ R^1, R^2 = Alk, Ar, Het; X = H, $SiMe_3$.	$Pd - CuI - PPh_3$, KF/Al_2O_3, MW, 2.5 min	26	31 – 98	58
6	$HC{\equiv}CSiMe_3 + ArX \longrightarrow ArC{\equiv}CSiMe_3$ X = Hal, OTf.	$Pd(PPh_3)_2Cl_2$, CuI, Et_2NH, DMF *a*) MW, 5 – 25 min *b*) Δ, 5 – 300 min	 26 6	 80 – 99 95 – 99	59
7	$RC{\equiv}CSiMe_3 \longrightarrow RC{\equiv}CH$	KF/Al_2O_3, MW, 1 – 3.5 min	17	0 – 98	60
8	$RC{\equiv}CH \longrightarrow RC{\equiv}C{-}C{\equiv}CR$ R = Alk, Ar.	KF/Al_2O_3, $CuCl_2$, MW, 8 min	7	53 – 75	61
9	$RC{\equiv}CR + PhC{\equiv}CPh \longrightarrow RC{\equiv}CPh$ R = Alk, $SiMe_3$.	$Mo(CO)_6$, n-C_8H_{18}, MW, 10 – 15 min	7	~50	62
10	$PhC{\equiv}CH + RCOCl \longrightarrow PhC{\equiv}C{-}\underset{\underset{O}{\|}}{C}{-}R$ R = Alk, Ar.	CuI, Et_3N *a*) MW, 10 min *b*) Δ, 30 h $Pd(PPh_3)_2Cl_2/CuI$, Et_3N, KF/Al_2O_3 *a*) MW, 4 min *b*) Δ, 8 h	 9 9 7 7	 76 – 93 52 – 81 81 – 94 72 – 90	63 64

cross-coupling (reactions Nos 5–7), aromatic nucleophilic (Nos 8–21) and electrophilic (No. 22) substitution reactions.

The Diels–Alder reactions yielding aromatic compounds (Nos 1–4) manifest prominent MWE. In particular, in example No. 1, microwave activation gives a significant improvement in the yields of reaction products at constant reaction times as compared to thermal activation. In example No. 2, the application of MWI substantially accelerates the reaction and increases the yields by 20%. The synthesis of quinoxalines (reactions Nos 3 and 4) from non-activated dienophiles can be realised only under solvent-free MWI conditions. Normally, such reactions require heating up to 200 °C, but the intermediate product, quinodimethane, rapidly decomposes at this high temperature and thus quinoxalines are produced only in trace amounts (< 3%).

The microwave-assisted Suzuki cross-coupling reactions are complete in 1–15 min giving yields as high as 40%–98%. Unfortunately, no results for activation methods other than MWI have been reported in those cases, thus no conclusion on the extent of the MWE can be deduced.

The use of MWI for the activation of aromatic nucleophilic substitution reactions is not as efficient. In only a few cases, slight acceleration of the reactions (*e.g.*, Nos 8 and 10) and an increase in the yields of target products by 10%–20% were registered. An exception to this is the substitution of the hetarylthio group for the chlorine atom in the aromatic core (No. 21). In this specific case, the reaction rate increased ~140-fold in the presence of a solvent and 480-fold when the reaction was conducted in the absence of solvents on a solid support, which is plain evidence for the MWE.

Table 4. Synthesis of aromatic compounds and reactions with them.

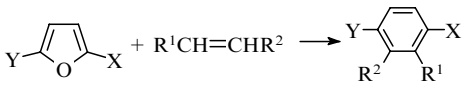
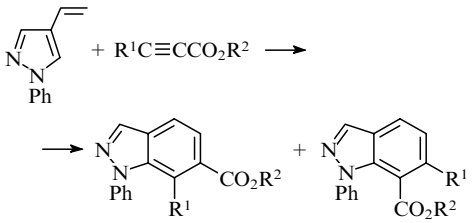
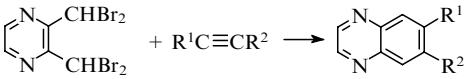
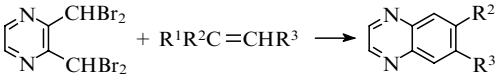
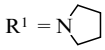
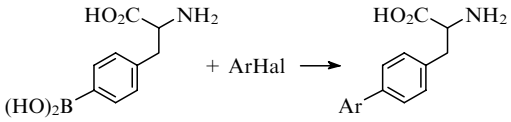
No.	Reaction	Conditions	Number of examples	Yield (%)	Ref.
Synthesis of aromatics					
1	 X = Me, Et, OMe; Y = H, Me; R^1 = CN, CO_2Me; R^2 = H, CN.	*a*) Si(M) (M = Ti, Zn, Al), MW, 25–45 min	25	0–100	65
		b) Δ, 25–45 min	8	3–31	65
2	 R^1 = H, CO_2R^2, Ph; R^2 = Me, Et.	*a*) MW, 6–30 min	4	22–72 [a]	66
		b) CH_2Cl_2, Δ, 3–7 days	4	7–53 [a]	66
3	 R^1 = H: R^2 = Ph, 1-Naph; R^1 = Me, R^2 = Ph.	NaI, DMF, MW, 13–15 min	3	38–43	67
4	 R^1 = NO_2, R^2 = H, R^3 = Ph; R^1 = , R^2–R^3 = $(CH_2)_n$ (n = 3, 4).	NaI, DMF, MW, 10–15 min	3	33–69	67
Cross-coupling reactions of aromatic compounds					
5	HetHal + Ph_2BNa ⟶ HetPh	$Pd(OAc)_2$, Na_2CO_3, H_2O or MeNHCHO, MW, 8–12 min	7	60–85	68
6	$Ar^1B(OH)_2$ + Ar^2Hal ⟶ Ar^1—Ar^2	*a*) $PdCl_2$, KF, PEG-400, MW, 50 s	16	66–92	69
		b) $Pd(OAc)_2$, KF/Al_2O_3, MW 2–15 min	6	40–98	31
		c) $Pd(OAc)_2$, Na_2CO_3, Bu^n_4NBr, H_2O, MW, 5 min	25	45–96	70
7		$Pd(PPh_3)_2Cl_2$, Na_2CO_3, $MeCN–H_2O$, MW, 5–10 min	9	68–86	71

Table 4 (continued).

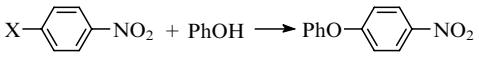
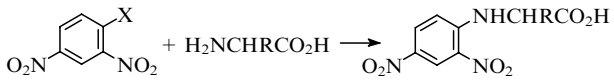
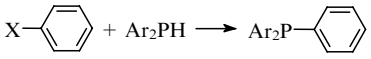
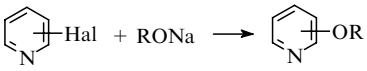
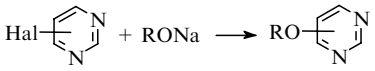
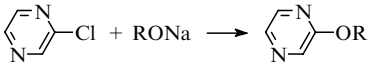
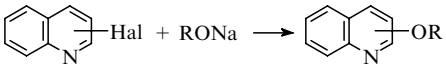
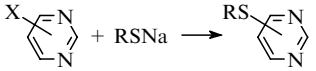
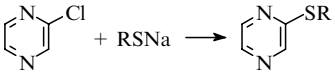
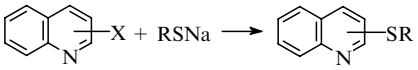
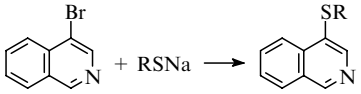
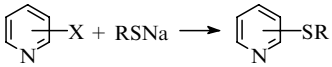
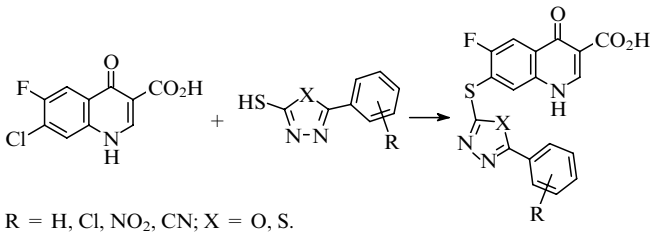
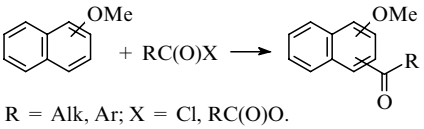
No.	Reaction	Conditions	Number of examples	Yield (%)	Ref.
Aromatic nucleophilic substitution reactions					
8	 X = Cl, F.	*a*) KOH, $R_4N^+Y^-$ or 18-crown-6, MW, 0.5–10 min	15	38–98	72
		b) Δ, 0.5–10 min	14	24–86	72
9	 R = H, Alk, Ph; X = F, Cl, Br.	$NaHCO_3$, H_2O, MW, 0.6–15 min (N-arylation)	18	48–93	73
10	ArX + MeOK ⟶ ArOMe X = F, Cl.	*a*) $R_4N^+Y^-$ or 18-crown-6, MW, 3.5–90 min	15	38–100	72
		b) Δ, 3.5–1440 min	9	24–93	72
11	 X = I, Br, OTf.	Al_2O_3, $Pd(OAc)_2$ or Pd/C or $Pd(PPh_3)_2$, AcOK, DMF, MW, 2–20 min	18	26–98	74
12	 R = Ph, Bn.	NMP [b] or HMPA,[c] or DMSO, MW, 45–900 s	28	0–95	75
13	 R = Ph, Et.	NMP or HMPA, or DMSO, MW, 35–600 s	11	20–88	76
14	 R = Ph, Et.	NMP or HMPA, or DMSO, MW, 40–50 s	4	54–69	76
15	 R = Me, Et; Hal = Cl, Br.	NMP or HMPA, MW 30–360 s	12	30–99	77
16	 R = Me, Ph; X = Cl, Br.	NMP or HMPA, or DMSO, MW, 30–60 s	15	7–97	76
17	 R = Me, Ph.	NMP, MW, 35–50 s	2	88–96	76
18	 R = Me, Ph; X = Cl, Br.	NMP or HMPA, MW, 30–240 s	17	0–99	77
19	 R = Me, Ph.	NMP or HMPA, MW, 1–10 min	5	0–98	77
20	 R = Me, Ph; X = Hal.	NMP or HMPA, MW, 0.5–10 min	28	0–99	78

Table 4 (continued).

No.	Reaction	Conditions	Number of examples	Yield (%)	Ref.
	Aromatic nucleophilic substitution reactions				
21	 R = H, Cl, NO_2, CN; X = O, S.	*a*) Al_2O_3, MW, 60–90 s *b*) CH_2Cl_2, MW, 4–5 min *c*) Δ, 10–12 h	10 10 10	90–96 70–78 59–65	79 79 79
	Aromatic electrophilic substitution reaction				
22	 R = Alk, Ar; X = Cl, RC(O)O.	EPZG®,[d] MW, 2–5 min (X = Cl), 4–8 min (X = RC(O)O)	12	38–100[a]	80

[a] The total yield of isomers is given. Hereinafter, [b] NMP is *N*-methylpyrrolidone-2; [c] HMPA is hexamethylphosphortriamide, [d] EPZG is a support material.

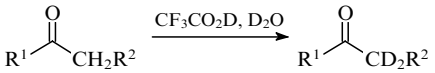
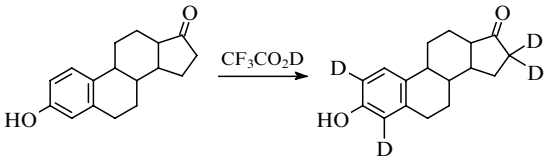
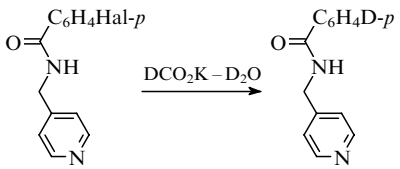
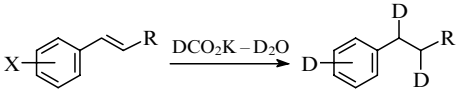
V. Deuterated derivatives

Examples of the use of MWI for the introduction of deuterium labels through the substitution of a hydrogen (examples Nos 1 and 2) or halogen (No. 3) atom with deuterium or addition of deuterium to a double bond are given in Table 5. These data imply that neither microwave nor thermal activation is sufficient to complete the deuteroexchange, although the reaction proceeds somewhat faster in the former mode.

VI. Synthesis of alcohols and phenols. Deprotection of the OH group

Studies reporting the application of MWI to the synthesis of alcohols and phenols are quite numerous. Most frequently, alcohols are produced by the reduction of aldehydes, ketones or esters with Pr^iOH or $NaBH_4$ (reactions Nos 1–3, Table 6). In addition to reduction, the hydrolysis of esters (reaction No. 4) and opening of oxirane rings (reaction No. 5) are utilised. The

Table 5. Microwave-assisted introduction of deuterium labels.

No.	Reaction	Conditions	Number of examples	Yield (%)	Ref.
1	 R^1 = Me, biphenyl; R^2 = Alk.	*a*) MW, 15–20 min *b*) Δ, 8–24 h	5 5	75–96 68–90	81 81
2		*a*) EtOH, MW, 10–600 s *b*) Δ, 1–1200 min	9 6	0–95 2–92	82 82
3		EtOH or DMSO, Pd/C or $Pd(OAc)_2$, MW, 20 s	3	> 50	83
4	 X = Cl, Br; R = H, CO_2H.	$RhCl_3$, $Pd(OAc)_2$, DMSO, MW, 40 s	3	> 95	84

hydroxymethylation of uracils in basic media (reaction No. 6) and controlled Cleisen rearrangement (No. 7) have also been reported.

The above reactions under MWI are complete in a few minutes affording the respective hydroxy-containing products in yields as high as 90% – 99%. In particular, the reaction times of the Claisen rearrangement (No. 7) and benzaldehyde reduction (No. 1) were reduced by factors of 28 and 84, respectively, as compared to the thermally activated reactions, although the product yields remained essentially unchanged.

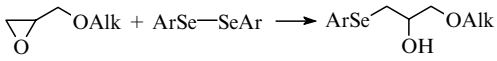
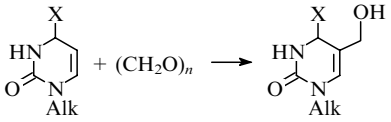
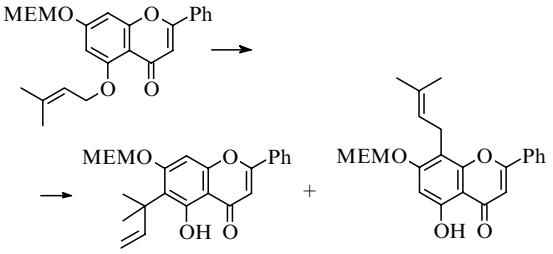
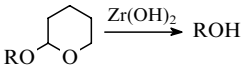
Several authors reported the microwave-assisted deprotection of OH groups (reactions Nos 8 and 9 in Table 6).

VII. Reactions involving alcohols and phenols

Examples of chemical modifications of alcohols assisted by MWI given in Table 7 include substitution (No. 1), O-sulfonylation (No. 2), O-alkylation (Nos 3 – 11), O-glycosylation (Nos 12 and 13) and protection of the OH group (Nos 14 – 17). These data indicate that in some cases, the MWI reduces the reaction times and increases the yields of products as compared to the reactions under ordinary heating. For instance, the yields of 3,4-dialkoxybenzaldehydes (reaction No. 9) increased from 60% in the classical synthesis (K_2CO_3, boiling acetone) to 86% in the solvent-free MWI synthesis under conditions of phase-transfer catalysis with a simultaneous reduction of the reaction times from 1440 min to 10 min. The use of MWI proved promising in the opening of the aziridine ring with alcohols (reaction No. 11). The reaction time in this case was reduced by a factor of 480 as compared to the thermally activated reaction. Furthermore, the opening of the aziridine ring with phenol appeared possible only under the MWI conditions. This is a clear manifestation of the MWE.

Nevertheless, the use of MWI does not give positive results in all cases. For instance, the microwave-assisted O-alkylation (No. 3) proceeds 2 times slower than the thermally activated

Table 6. Synthesis of alcohols and phenols. Deprotection of the OH group.

No.	Reaction	Conditions	Number of examples	Yield (%)	Ref.
Synthesis of alcohols					
1	$PhCHO + (CH_2O)_n \longrightarrow PhCH_2OH$	*a*) KF/Al_2O_3, MW, 5 min	1	92	85
		b) Δ, 7 h	1	85	85
2	$R^1COR^2 \xrightarrow{Pr^iOH} R^1CH(OH)R^2$	*a*) Ru catalyst, MW, 1.5 – 14 min	19	24 – 99	86
	R^1 = Ph: R^2 = Me, Bu^t (conditions *a*); R^1 = Alk, Ar; R^2 = H, Alk, Ar (conditions *b*).	*b*) Al_2O_3, KOH, MW, 9 – 16 min	28	85 – 93	87
3	$R^1CO_2R^2 \longrightarrow R^1CH_2OH + R^2OH$	$NaBH_4/LiCl$, MW, 2 – 8 min	12	55 – 97	88
	R^1 = Ar, Bn; R^2 = Me, Et.				
4	$R^1CO_2R^2 + H_2O \longrightarrow R^1CO_2H + R^2OH$	KF/Al_2O_3, MW, 2 min	7	92 – 97	89
	R^1 = Ph, Me; R^2 = Alk, Ph, Bn.				
5		$NaBH_4$, NaOH – EtOH, MW, 13 min	11	84 – 92	90
6		KOH, MW, 3 min	6	93 – 99	91
	X = O, NH_2.				
7		*a*) $PhEt_2N$, MW, 30 – 150 min	4	83 – 90 [a]	92
		b) Et_2Bu^nN, Δ, 72 h	1	86 [a]	92
	MEM = $MeOCH_2CH_2OCH_2$.				
Deprotection					
8	$ROSiMe_3 + H_2O \longrightarrow ROH$	kaolinite, MW, 1 – 2.5 min	17	58 – 87	93
	R = Alk, Ar.				
9		*a*) MW, MeCN, 7 – 10 min	16	80 – 97	94
		b) Δ, 65 – 95 min	16	70 – 90	94
	R = Alk, Ar.				

[a] The total yield of products is given.

Table 7. Reactions involving alcohols and phenols.

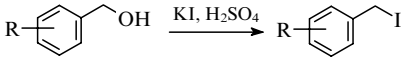
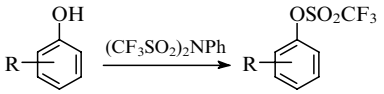
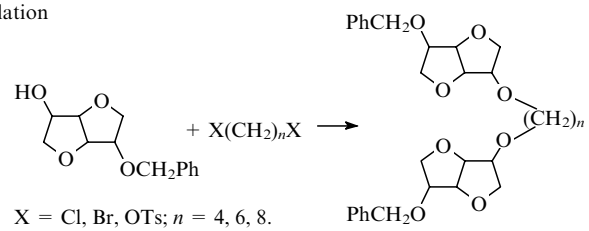
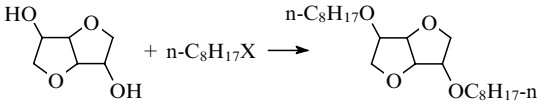
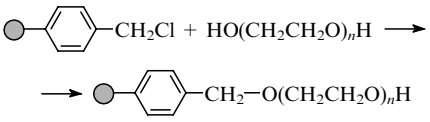
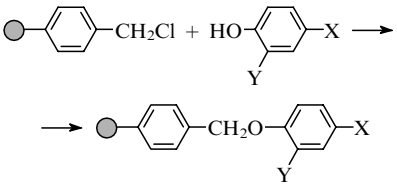
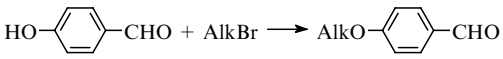
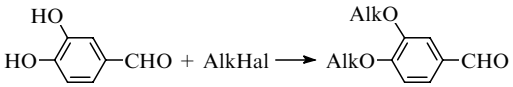
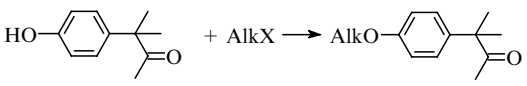
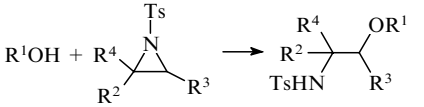
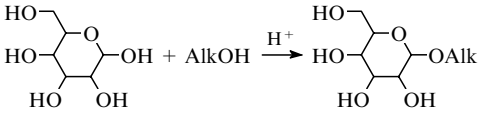
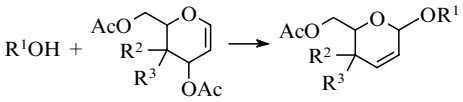
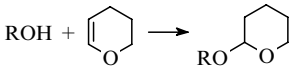
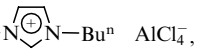
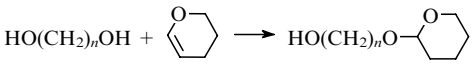
No.	Reaction	Conditions	Number of examples	Yield (%)	Ref.
Substitution reactions					
1	 R = H, Hal, NO_2.	clay, MW, 2–8 min	15	74–93 [a]	95
O-Sulfonylation					
2		K_2CO_3, THF, MW, 6 min	10	69–91	96
O-Alkylation					
3	 X = Cl, Br, OTs; n = 4, 6, 8.	KOH, Bu_4^nNBr, PhMe or xylene, or cyclohexane			97
		a) MW, 5–30 min	31	27–96	
		b) Δ, 15 min (for X = OTs)	10	36–92	
4	 X = Cl, Br, OTs, OMs, OSO_2R.	*a*) MOH (M = Li, Na, K, Cs), Bu_4^nNI, xylene, MW, 10 min	40	0–96	98
		b) Δ, 10 min	39	0–76	98
5	ArOH + RCl ⟶ ArOR R = Bn, Ar.	$NaOH-H_2O$, MW, 1–2.5 min	9	52–88	99
6	 n = 5–23.	*a*) NaOH, MW, 2 min	9	34–39	100
		b) Δ, 30 min	5	40–60	100
7	 X = CHO, CO_2Et; Y = H, OMe.	MeONa, DMF–EtOH, MW, 7 min	3	82–95	101
8		KOH, DMF–benzene			102
		a) MW, 45–60 min	2	85–96	
		b) Δ, 6 h	2	58–62	
9	 Alk = C_nH_{n+1} (n = 12, 14, 16, 18).	*a*) KOH + K_2CO_3, Bu_4^nNBr, MW, 10 min	4	80–86	103
		b) K_2CO_3, Me_2CO, Δ, 1440 min	4	49–66	103
10	 X = Hal, SAlk.	anion-exchange resin, MW, 3–5 min	10	62–90	104
11	 R^1, R^4 = Alk, Ar; R^2, R^3 = H, Alk; R^2-R^3 = $(CH_2)_n$ (n = 3, 4).	$Sn(OTf)_2$ or $BF_3 \cdot Et_2O$			105
		a) MW, 15 min	39	40–98	
		b) Δ, 5–7200 min	39	0–99	

Table 7 (continued).

No.	Reaction	Conditions	Number of examples	Yield (%)	Ref.
O-Glycosylation					
12		MW, 4–60 min	20	10–100	106
13	 R^1 = Alk, Ar; R^2, R^3 = H, OAc.	*a*) K-10,[b] MW, 10–20 min *b*) K-10,[b] PhCl, MW, 2–15 min	6 20	71–87 52–93	107 108
Protection of the OH group					
14	 R = Alk, Ar.	*a*) $Zr(OH)_2$, MW, 7–15 min *b*) Δ, 50–110 min *c*)  MW, 15–30 min *d*) $FeSO_4$, MW, 0.5–4 min	20 21 9 13	81–97 70–91 74–98 78–97	94 94 109 110
15	 *n* = 2–6.	I_2, THF, MW, 150–170 s	5	75–78[c]	78
16	$ROH + Pr^i_3SiCl \longrightarrow ROSiPr^i_3$ R = Alk, Ar.	imidazole, MW, 30–120 s	14	70–96	111
17	$ROH + (Me_3Si)_2NH \longrightarrow ROSiMe_3$ R = Alk, Ar.	$LiClO_4$, MW, 1–4 min	17	58–83	93

[a] The reaction is impossible with aliphatic alcohols and phenols; [b] Hereinafter, K-10 is a montmorillonite modification; [c] the yield of the disubstituted product is 15%–17%.

reaction. In a few cases, the yields of MWI reactions were lower than those of the thermal reactions, despite some acceleration (No. 6).

VIII. Synthesis of aldehydes and ketones. Protection and deprotection of the carbonyl group

Among methods used for the microwave-assisted synthesis of carbonyl compounds, the most common are: oxidation of an activated methylene group (Table 8, reaction No. 1); selective oxidation of alcohols [in particular, oxidation of benzyl alcohols with calcium hypochlorite (No. 2), mild oxidation of benzoin over alumina or zeolites without oxidants (No. 3) and oxidation of 1-hydroxyphosphonates on solid supports (No. 4)]; selective dealkylation (No. 5); oxidative deprotonation of tetrahydropyranyl (No. 6) or trimethylsilyl ethers (No. 7), the Heck reaction (No. 8), intramolecular condensation of dicarboxylic acids over graphite (No. 9), condensation of carboxylic acid chlorides with 2-methylquinolines (No. 10), cross-coupling of sodium organoborates with acyl and aroyl chlorides (No. 11), catalytic [2 + 2 + 1]-cycloaddition (the Pauson–Khand reaction, see Nos 17 and 18 in Table 2).

Table 8. Synthesis of aldehydes and ketones.

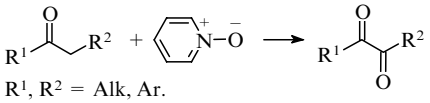
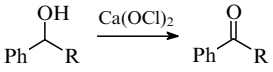
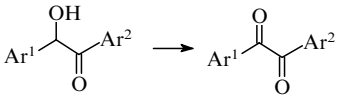
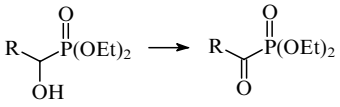
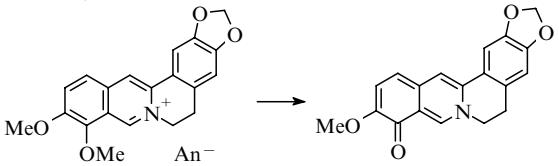
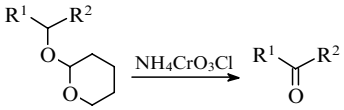
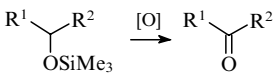
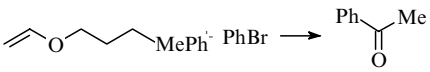
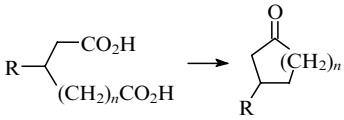
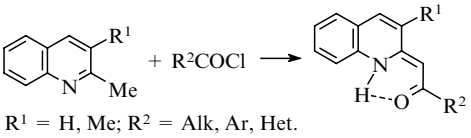
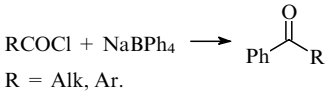
No.	Reaction	Conditions	Number of examples	Yield (%)	Ref.
1	 R^1, R^2 = Alk, Ar.	HNIB,[a] MW, 90–150 s	10	45–90	112
2	 R = H, Alk.	H_2O, Al_2O_3, MW, 6–120 s	8	57–98	113

Table 8 (continued).

No.	Reaction	Conditions	Number of examples	Yield (%)	Ref.
3		*a*) Al_2O_3, MW, 1 min	1 [b]	65	113
		b) zeolite A, MW, 6 min	6	69–91	114
4	 R = Alk, Ar.	Cr_2O_3/Al_2O_3, MW, 0.5–3 min	17	68–89	115
5		Al_2O_3, MW, 5 min	1	98	116
6	 R^1 = Alk, Ar; R^2 = H, Alk.	K-10, MW, 20–120 s	10	78–94	117
7	 R^1 = Alk, Ar; R^2 = H, Alk; [O] — zeofen [c] (*a*), $[PhCH_2PPh_3][HSO_5]$ (*b*).	*a*) MW, 20–120 s	9	78–98	118
		b) $BiCl_3$, CH_2Cl_2, MW, 3–5 min	10	80–99	119
8		*a*) 1) DMF–H_2O, MW, 1 h; 2) H^+	1	62	120
		b) [Pd], $Ph_2P(CH_2)_3PPh_2$ (dppp), K_2CO_3, DMF–H_2O (12.5 : 1), 80 °C, 16 h	1	82	120
9	 R = H, Me; *n* = 2–4.	*a*) graphite, Fe, Al or Bi oxides, MW, 4 min	11	10–90	121
		b) Δ, 30 min	6	7–90	121
10	 R^1 = H, Me; R^2 = Alk, Ar, Het.	*a*) SiO_2, MW, 4 min	13	53–91 [d]	122
		b) 200 °C, 60 min	13	0	122
11	 R = Alk, Ar.	*a*) $Pd(PPh_3)_2Cl_2$, KF, Me_2CO, MW, 9 min	10	87–98	123
		b) $Pd(PPh_3)_2Cl_2$, KF/Al_2O_3, Me_2CO MW, 5 min	10	90–97	124
		c) Δ, 20 h	10	68–90	123

[a] HNIB is hydroxy(*p*-nitrobenzylsulfonyl)iodobenzene; [b] for $Ar^1 = Ar^2 = Ph$; [c] zeofen is $Fe(NO_3)_3$/HZSM-5 oxidant; [d] the reaction is impossible for carboxylic acid anhydrides with $R^2 = Bu^t$ and Pr^i.

A comparison of the microwave and thermal methods for the activation of these reactions indicate that the yields of target products are virtually identical in both cases, but the use of MWI accelerates the reactions. Furthermore, the condensation of carboxylic acid chlorides with 2-methylquinolines (No. 10) is possible only under the MWI conditions.

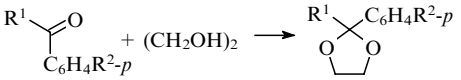
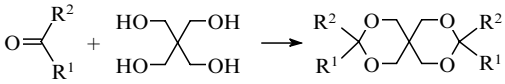
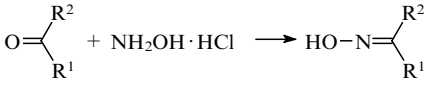
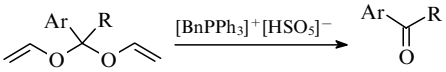
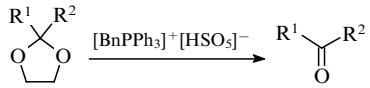
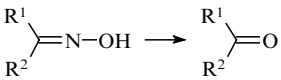
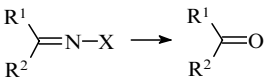
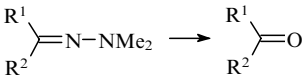
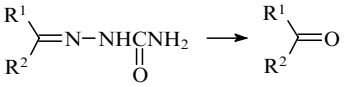
Acid anhydrides (Table 9, No. 1), polyatomic alcohols (Nos 2 and 3) or hydroxylamine (No. 4) are commonly used to protect the carbonyl groups in various chemical transformations.

As deprotection methods, the oxidative deprotonation of acetals and ketals (Nos 5 and 6), mild oxidative conversion of oximes (Nos 7 and 8), hydrazones (Nos 8 and 9) and semicarbazones (No. 10) over various supports (*e.g.*, K-10, SiO_2, Al_2O_3, *etc.*) are utilised.

IX. Synthesis of carboxylic acids, esters and lactones

Table 10 summarises examples of the utilisation of MWI in the synthesis of carboxylic acids. The hydrolysis of esters over solid supports (No. 1) and amides in the presence of sulfuric acid with variable concentrations (No. 2) and the so called Mathews dry

Table 9. Microwave-assisted protection and deprotection of the carbonyl group.

No.	Reaction	Conditions	Number of examples	Yield (%)	Ref.
Protection of the carbonyl group					
1	$RCHO + Ac_2O \longrightarrow RCH(OAc)_2$	*a*) envirocat EPZ10,[a] MW, 10–55 s	13	83–92	125
		b) $FeCl_3/SiO_2$, MW, 3–5 min	12	78–95	126
2		*a*) H^+, benzene, MW, 50–120 min	4	75–90	102
		b) Δ, 4–5 h	4	58–72	102
	R^1 = H, Me; R^2 = Alk.				
3		$H_3[PW_{12}O_{40}]$, MeCN, MW, 2–12 min	10	0–93	127
	R^1 = Alk, Ar; R^2 = H, Alk, Ar.				
4		*a*) Al_2O_3, MW, 1–7 min	18	47–99	128
		b) molecular sieves, 20 °C, 30–95 min	18	0–88	128
	R^1 = Alk, Ar; R^2 = H, Me.				
Deprotection of the carbonyl group					
5		$BiCl_3$, CH_2Cl_2, MW, 4–6 min	6	80–95	119
	R = H, Alk.				
6		$BiCl_3$, CH_2Cl_2, MW, 3–5 min	10	80–96	119
	R^1 = Alk, Ar; R^2 = H, Alk.				
7		*a*) K-10, MW, 1–6 min	7	80–94	129
		b) BTSC[b]/K-10, MW, 60 min	7	88–93	130
		c) BTSC[b]/K-10, CH_2Cl_2, MW, 40 min	7	84–90	130
	R^1 = Alk, Ar; R^2 = H, Alk, Ar.	*d*) HCO_2H/SiO_2, MW, 2–4 min	13	86–94	131
8		zeolite-41/SiO_2, (see[c]), Cr-MSM-4/SiO_2 (see[c]), MW, 5–10 min	30	72–98	132
	R^1 = Alk, Ar; R^2 = H, Alk, Ar; X = OH, NHTs.				
9		*a*) $CeCl_3 \cdot 7H_2O$, SiO_2, MW, 5–10 min	19	83–91	133
	R^1 = Alk, Ar; R^2 = H, Alk, Ar.	*b*) $PdCl_2 \cdot SnCl_2 \cdot H_2O$, MW, 90 s	8	78–98	134
10		*a*) $Fe(NO_3)_3 \cdot 9H_2O$ + K-10 (clayfen), MW, 20–240 s	11	82–95	135
	R^1 = Alk, Ar; R^2 = H, Alk, Ph.	*b*) CAN[d]/Al_2O_3, MW, 10–300 s	11	68–90	136

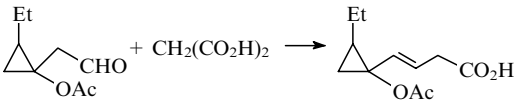
[a] Envirocat EPZ10 is a support material; [b] BTSC is bis(trimethylsilyl)chromate; [c] Cr-MSM-4 is an ion-exchange aluminosilicate; [d] CAN is cerium ammonium nitrate.

hydrolysis of amides and nitriles in the presence of phthalic acid under the solvent-free conditions (Nos 3 and 4) are the most common routes. β,γ-Unsaturated carboxylic acids can be prepared by Knoevenagel condensation of aldehydes with maleic acid followed by double bond isomerisation (No. 5). For the latter reaction, the use of solvent-free MWI conditions not only reduced the reaction time but also increased the yield of the product by 43%, which is evidence of the MWE.

Carboxylic acid esters can be prepared under MWI conditions by the interaction of acids with alcohols (Table 11, Nos 1–3). Polyesters are formed in the intermolecular polycondensation of L-2-hydroxy-3-phenylpropionic acid (No. 4) or catalytic polymerisation of lactones (No. 5).

Several examples of solid-state microwave synthesis of esters (Nos 6 and 7) by the interaction of aliphatic or aromatic acids with *para*-substituted benzyl chloride grafted to a solid support or

Table 10. Synthesis of carboxylic acids.

No.	Reaction	Conditions	Number of examples	Yield (%)	Ref.
1	$RCO_2Alk \xrightarrow{H_2O} RCO_2H$ R = Alk, Ar.	KF/Al_2O_3, MW, 2 min	14	90–98	89
2	$PhCONH_2 \longrightarrow PhCO_2H$	*a*) 20% H_2SO_4, MW, 1–7 min	9	25–99	137
		b) 5% H_2SO_4, MW, 1–10 min	13	9–99	137
3	$RCONH_2 \xrightarrow{C_6H_4(CO_2H)_2\text{-}o} RCO_2H$ R = Alk, Ph.	MW, 10 atm, 15–30 min	7	53–99	138
4	$RC{\equiv}N \xrightarrow{C_6H_4(CO_2H)_2\text{-}o} RCO_2H$ R = Alk, Ph.	MW, 10 atm, 15–30 min	3	98–99	138
5		*a*) SiO_2, MW, 15 min	1	83	139
		a) Δ, xylene, 2 h	1	40	139

benzoic acid anhydride with support-grafted alcohols were reported.

Furthermore, the selective benzoylation of the primary alcohol group in 1,2-dihydroxyalkanes (No. 8) and of the keto group in pyrazolone (No. 9), the catalytic acetylation of sterically hindered phenols and 5-hydroxyflavones with acetic anhydride (No. 10) and the esterification of acids with dimethyl carbonate (No. 11) were accomplished. These reactions were conducted both under thermal and MWI conditions. According to the results obtained, the use of MWI reduces the reaction times significantly.

Table 11. Synthesis of esters and lactones.

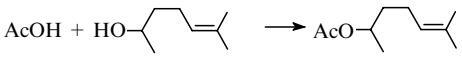
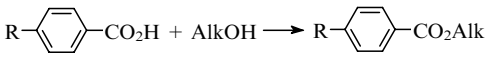
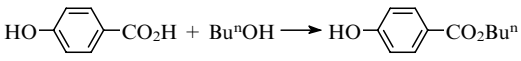
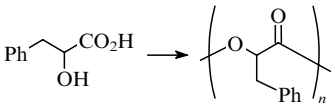
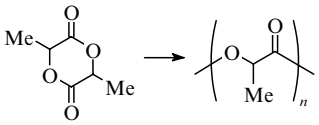
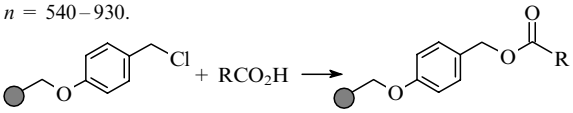
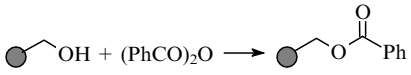
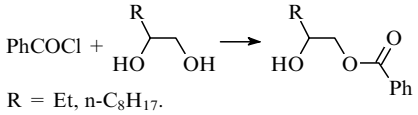
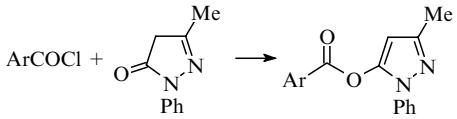
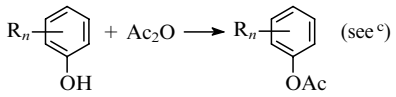
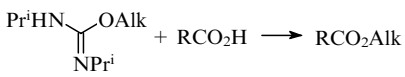
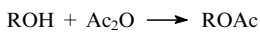
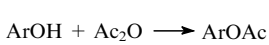
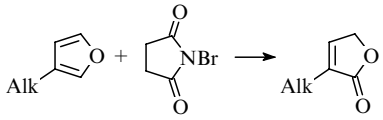
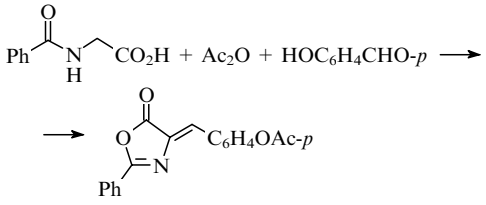
No.	Reaction	Conditions	Number of examples	Yield (%)	Ref.
Synthesis of esters					
1		PPh_3, $Pr^iO_2CN{=}NCO_2Pr^i$, MW, 5–7 min	5	72–99	140
2	 R = H, OH.	MW, 22–60 min	8	76–90	141
3		*a*) $ZnCl_2$, MW, 150 s	1	43	142
		b) TsOH, MW, 150 s	1	41	142
		c) $ZnCl_2$, Δ, 45 min	1	3.5	142
		d) TsOH, Δ, 45 min	1	76	142
4	 *n* = 25–55.	*a*) MW, 1.5–2.5 h	2	20–22	143
		b) Δ, 30–96 h	2	36–38	143
5	 *n* = 540–930.	$(n\text{-}C_8H_{17})_2SnO$, MW, 10–60 min	5[a]	31–57	144
6	 R = Alk, Ar.	Cs_2CO_3, MW, 3–15 min	33	73–99	145

Table 11 (continued).

No.	Reaction	Conditions	Number of examples	Yield (%)	Ref.
Synthesis of esters					
7		NMP, MW, 5–20 min	10	27–99	146
8	 R = Et, n-C_8H_{17}.	*a*) Bu^n_2SnO, Et_3N, PhMe, MW, 9 min *b*) Δ, 7–820 min	9 5	29–99 61–95 [b]	147 147
9		MW, 8 min	8	37–87	148
10	 (see [c]) R_n = 2-Ac; 2,6-Bu^t_2-4-X (X = OH, CO_2H); 2-$C(O)CH_2OMe$-3,5-$(OH)_2$.	*a*) I_2, MW, 2–4 min *b*) Δ, 3–5 h	7 7	94–98 50–70	149 149
11	 R = Alk, Ar.	*a*) DBU,[d] MW, 2–24 min *b*) Δ, 4–24 h	9 8	95–99 90–99	150 150
12	 R = Alk, Bn.	THF, MW, 5 min	11	75–94	151
13	 R = Alk, Ar.	MW, 2–10 min	22	80–95	152
14		Al_2O_3, Py, MW, 1–6 min	13	54–100	153
Synthesis of lactones					
15		*a*) MW, 15–60 min *b*) ultrasound, 12–15 min *c*) Δ, 32–48 h	5 5 5	62–76 56–62 36–42	154 154 154
16		*a*) KF/$AlPO_4$,[e] MW, 15 min *b*) KF/Al_2O_3,[e] Δ, 4 h	25 25	0–87 0–72	155 155

[a] Different durations of irradiation and radiation power were used; [b] products of the substitution of the second OH group were also obtained; [c] the acetylation of substituted 5-hydroxyflavones was also accomplished; [d] DBU is 1,2-diazabicyclo[5.4.9]undec-7-ene; [e] 25 support types were elucidated, the support indicated provided the maximum yield of products.

The esterification of carboxylic acids can also be realised with O-alkylated isoureas (No. 12).

The acetylation of alcohols and phenols with acetic anhydride affords the respective acetic acid alkyl and aryl esters (Nos 13 and 14).

Lactones can be prepared by the mild oxidation of furan with *N*-bromosuccinimide (Table 11, reaction No. 15). The reaction was applied to the synthesis of naturally occurring biologically active limonoids. Gopalakrishnan *et al.*[154] reported the preparation of lactones under the conditions of thermal, ultrasound and microwave activation. The maximum yield was obtained with microwave activation, whereas the reaction time was the minimum in the case of ultrasound activation: it was 4 and 190 times shorter than those under microwave and thermal activations, respectively.

The three-component condensation of acetic anhydride, *p*-hydroxybenzaldehyde and hippuric acid afforded azlactones (No. 16).

X. Synthesis of amines, imines, oximes, substituted hydroxylamines, hydrazines and hydrazones and reactions with them

Table 12 lists examples of microwave-assisted syntheses of anilines (No. 1), imines (Nos 2 and 3), hydroxylamines (No. 4), hydrazines (Nos 5 and 6) and hydrazones (Nos 7 and 8). The yields of target products in these reactions were in the range of 81% – 99% and the reaction times were from 0.5 to 30 min.

For several of the above reactions, comparative results on the microwave and thermal conditions were reported, which indicated that the MWI gave rise to their substantial acceleration.

Among the microwave reactions of amines, the following classes are of note: reductive elimination of styrenes (Table 13, No. 1) and alkynes (No. 2), numerous reactions of N-alkylation (Nos 3 – 11) and N-arylation (Nos 12 – 20) and hydrogenation (No. 21).

According to the studies reporting comparative data (which are, unfortunately, rather sparse, see Table 13), the use of MWI somewhat accelerates the reactions but the respective improvement in the yields of products is only insignificant.

Table 12. Synthesis of amines, imines, substituted hydroxylamines, hydrazines and hydrazones.

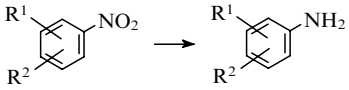
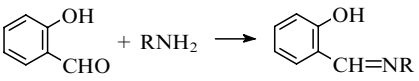
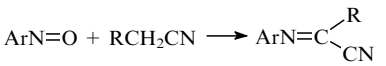
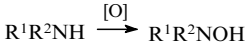
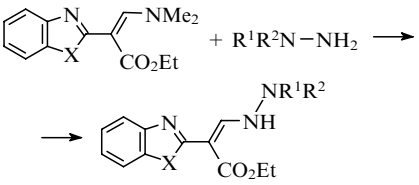
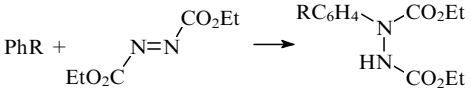
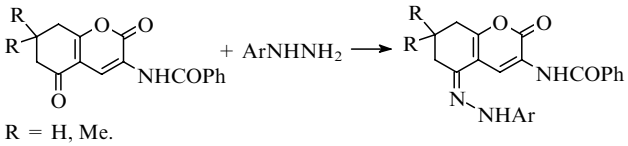
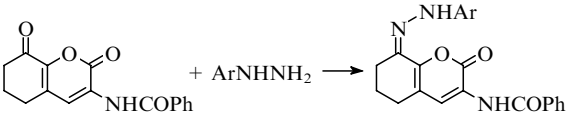
No.	Reaction	Conditions	Number of examples	Yield (%)	Ref.
1		*a*) NaH_2PO_4, $FeSO_4 \cdot 7H_2O$, MW, 50 – 100 s	15	68 – 88	156
	a) R^1, R^2 = H, Me, Ph, OH, NH_2, $CONH_2$, CO_2H, CN; *b*) R^1 = H, Hal, Me, OMe, OH, NH_2; R^2 = H, Alk.	*b*) $N_2H_4 \cdot H_2O/Al_2O_3$, $FeCl_3 \cdot 6H_2O$, MW, 8 – 10 min	11	81 – 97	157
2		MW, 0.5 – 4 min	16	77 – 98	158
	R = Ar, Het.				
3		LiBr, MW, 30 – 130 s	11	60 – 90	159
	R = Ar′, CN, CO_2H, CO_2Alk, COPh, $CONH_2$.				
4		OXONE, Al_2O_3 or SiO_2			160
	R^1 = Alk; R^2 = H, Alk.	*a*) MW, 40 – 60 s	5	89 – 97	
		b) Δ, 0.5 – 24 h	10	50 – 100	
5		*a*) MW, 30 min	5	90 – 95	161
		b) Δ, 6 – 216 h	3	71 – 80	161
	R^1 = H, Me; R^2 = Me, Ph, CO_2Me; X = O, S, NH.				
6		*a*) $InCl_3/SiO_2$, MW, 2 – 6 min	13	68 – 92	162
		b) Δ, 8 – 22 h	13	60 – 80	162
	R = Me, OMe.				
7		MW, 6 – 10 min	6	61 – 85	163
	R = H, Me.				
8		MW, 3 – 8 min	5	65 – 98	164

Table 13. Reactions involving amines.

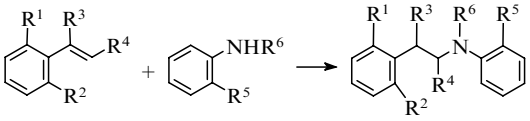
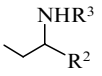
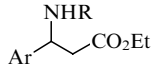
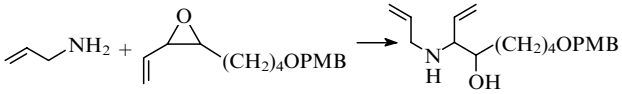
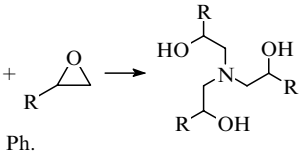
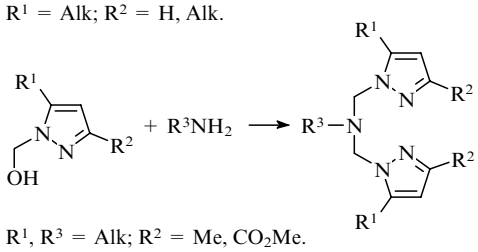
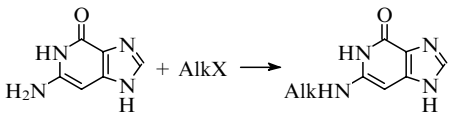
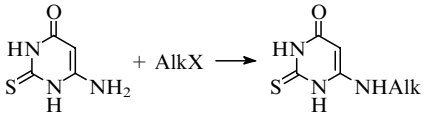
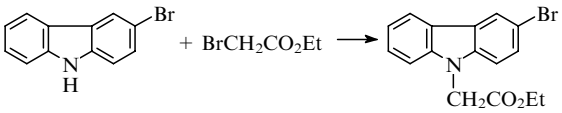
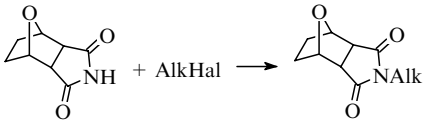
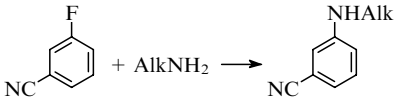
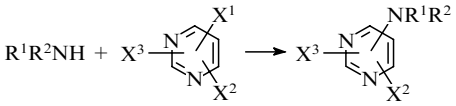
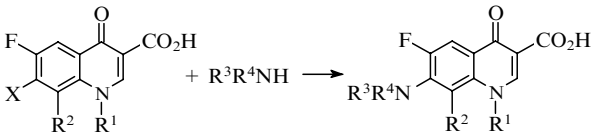
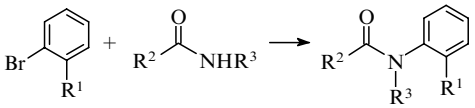
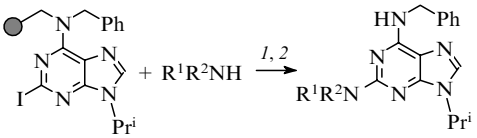
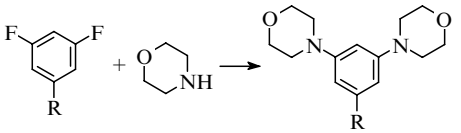
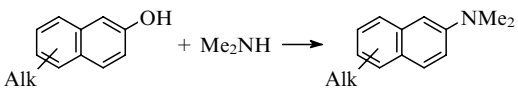
No.	Reaction	Conditions	Number of examples	Yield (%)	Ref.
Addition to multiple bonds					
1	 R^1-R^6 = H, Alk.	Bu^tOK, MW, 1.5–10 min	7	42–100	165
2	$R^1C{\equiv}CR^2 + R^3NH_2 \xrightarrow{1,\,2} R^1$ R^1-R^3 = Alk, Ar.	*1*) Cp_2TiMe_2, PhMe, MW, 2–3 h; *2*) H_2, Pd/C	18	34–93	166
N-Alkylation					
3	$RNH_2 + ArCHO + HO_2C(CH_2)CO_2Et \xrightarrow{AcOH}$ 	*a*) MW, 8–20 min	11	0–27[a]	167
		b) EtOH, Δ, 10–24 h	2	2–4	167
4	 PMB = *p*-$MeOC_6H_4CH_2$.	LiOTf, MeCN, MW, 1 h	1	92	168
5	$NH_4OH +$  R = Me, Ph.	*a*) AlkOH, MW, 35–180 min	8	43–100[b]	169
		b) Δ, 6 days	2	18–72[b]	169
6	$R^1R^2NH + HCHO \longrightarrow R^1R^2NMe$ R^1 = Alk; R^2 = H, Alk.	HCO_2H, DMSO, MW, 1 min	1	93	170
7	 R^1, R^3 = Alk; R^2 = Me, CO_2Me.	*a*) MW, 20 min	5	75–90	171
		b) Δ, 4 h	5	80–87	171
		c) MeCN, 25 °C, 4 days	5	80–85	171
8	 X = Cl, Br.	DMF, MW, 6–9 min	3	21–70	172
9	 X = Cl, Br.	*a*) DMF, MW, 9–11 min	2	83–84	172
		b) Na_2CO_3, Bu^n_4NI, MW, 6–10 min	9	87–95	172
10		KOH, DMF, MW, 2.4–6 min	8	52–85	173
11		K_2CO_3, Bu^n_4NBr, MW, 1–1.5 min	7	90–98	174

Table 13 (continued).

No.	Reaction	Conditions	Number of examples	Yield (%)	Ref.
N-Arylation					
12		*a*)  MW, 5 h	3	29 – 56	175
		b) Δ, 60 h	3	19 – 31	175
13	$R^1R^2NH + ArBr \longrightarrow R^1R^2NAr$ R^1, R^2 = H, Alk.	Pd catalyst, Bu^tONa, PhMe, MW, 4 – 15 min	13	0 – 86	176
14	$R^1C_6H_4Br\text{-}p + R^2R^3NH \longrightarrow R^1C_6H_4NR^2R^3\text{-}p$ R^1 = H, Me, OMe, CN; R^2, R^3 = H, Alk, Ph.	$Pd(OAc)_2$, Bu^tOK, DMF, MW, 4 min	13	48 – 85	177
15	 R^1 = Alk; R^2 = H, Alk; X^1 = Cl, Br; X^2 = H, Hal, OMe; X^3 = H, NH_2, OMe.	Pr^i_2EtN, Pr^iOH, MW, 5 – 160 min	12	29 – 97	178
16	 R^1, R^3, R^4 = Alk; R^2 = H, OAlk, F; X = F, Cl.	DMSO, MW, 8 – 35 min	9	75 – 94	179
17	 R^1 = H, OMe; R^2, R^3 = Alk, Ar.	CuI, K_2CO_3, NMP, MW, 20 – 40 min	6	51 – 77	180
18	 $R^1 = R^2 = HO(CH_2)_2$; R^1 = H, $R^2 = HO(CH_2)_3$.	*1*) NMP, MW, 30 min *2*) CF_3CO_2H, H_2O, 1 h	2	77 – 89	181
19	 R = CN, NO_2.	*a*) DMSO, MW, 5 h *b*) Δ, 60 h	2 2	65 – 76 16 – 37	175 175
20		H_2O, NH_3, $(NH_4)_2SO_3$, MW, 30 min	8	70 – 90	182
Hydrogenation					
21	$R^1R^2NX \longrightarrow R^1R^2NH$ R^1 = Alk, Ar; R^2 = Alk; X = Bn, Cbz.	HCO_2NH_4, Pd/C, Pr^iOH, MW, 3 – 8 min	14	70 – 95	183

[a] Ethyl esters of substituted cinnamic acids ArCH = $CHCO_2Et$ are also affforded in yields of 87% – 34%; [b] the monosubstitution products are formed in low yields.

XI. Synthesis of carboxylic acid amides and imides, their thio analogues, thioureas and thiosemicarbazides

Amides and imides can be synthesised by the microwave-assisted reactions of amines with carboxylic acids (or, alternatively, amine hydrochlorides with metal carboxylates) (Table 14, Nos 1 – 6) or with their derivatives, such as esters (No. 7), chlorides (Nos 8 and 9) or anhydrides (Nos 9 – 14). Other methods for the formation of amides include the specific Wolff rearrangement of α-diazoketones (No. 15), Heck aminocarbonylation of aryl bromides catalysed by palladium with DMF as a source of carbon oxide (No. 16) and hydrolysis of aromatic nitriles using different catalysts (No. 17). The preparation of amides from ketones and

Table 14. Synthesis of carboxylic acid amides and imides, their thio analogues, thioureas and thiosemicarbazides.

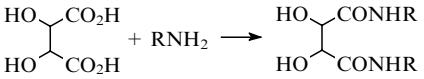
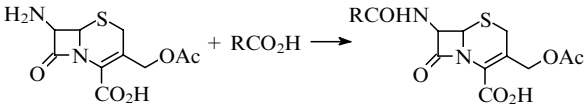
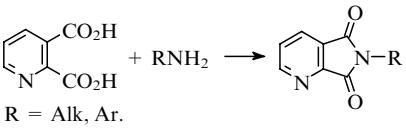
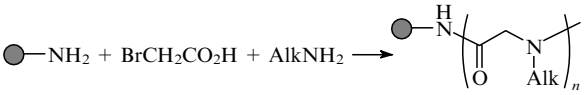
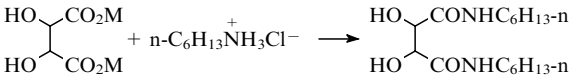
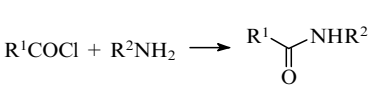
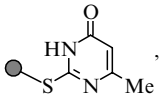
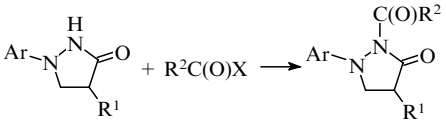
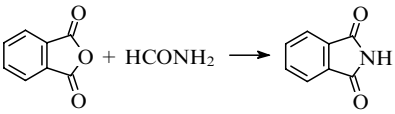
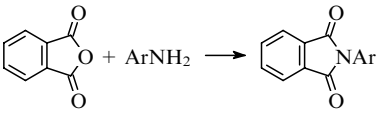
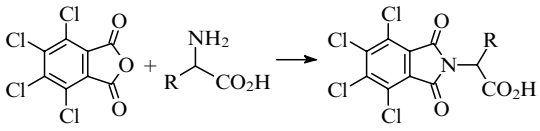
No.	Reaction	Conditions	Number of examples	Yield (%)	Ref.
1	$R^1CO_2H + R^2NH_2 \longrightarrow R^1CONHR^2$ R^1 = Alk, Ph; R^2 = Alk, Ar.	*a*) MW, 30–120 min *b*) Δ, 30–120 min	37 32	2–93 8–72	184 184
2	 R = Alk, Ph.	MW, 12 min	10	30–83	185
3	 R = Alk, Ar, Het.	*a*) Al_2O_3, MW, 90–120 s *b*) Δ, 2–6 h	10 10	82–93 63–76	186 186
4	 R = Alk, Ar.	*a*) Et_3N, MW, 90–300 s *b*) Δ, 0.5–2 h	11 5	51–95 0–20	187 187
5		*a*) DIC,[a] DMF, MW, 1 min *b*) Δ, 105 min	6 5	65–88 63–80	188 188
6	 M = Na, K.	MW, 25 min	2	13–40	185
7	$R^1CO_2R^2 + R^3R^4NH \longrightarrow R^1CONR^3R^4$ R^1 = Alk; R^2 = Me, Et: R^3, R^4 = H, Alk.	*a*) Bu^tOK, MW, 2–8 min *b*) Δ, 3–48 h	20 15	17–97 3–42	189 189
8	 R^1 = Me, Ph, *p*-XC_6H_4 (X = Me, Cl, Br), Bn, PhCH=CH, *o*-BrC_6H_4; R^2 = Bn, $(CH_2)_4OH$.	, Py, MW, 3 min	9	42–100	190
9	 R^1 = H, Me; R^2 = Alk, Ar; X = Cl, $OC(O)R^2$.	MW, 1 h	11	19–63	191
10	$R^1R^2NH + Ac_2O \longrightarrow AcNR^1R^2$ R^1 = Alk, Ar; R^2 = H, Alk.	Al_2O_3, MW, 0.25–16 min	36	65–100	152
11		MW, 2 min	1	98	192
12		MW, 2–10 min	8	91–95	193
13	 R = Alk, Ar.	*a*) MW, 4–8 min *b*) Δ, 24.5 h	30 6	62–69 67–98	194 194

Table 14 (continued).

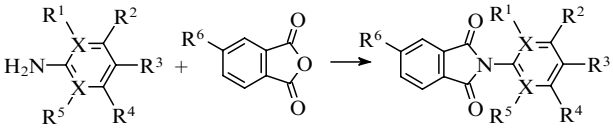
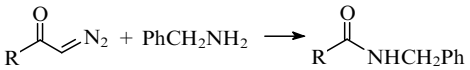
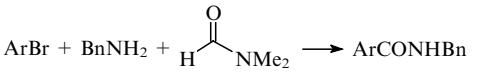
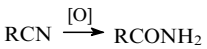
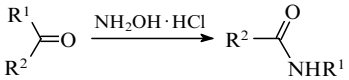
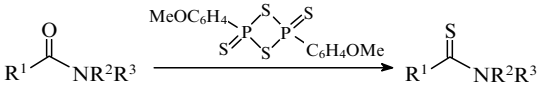
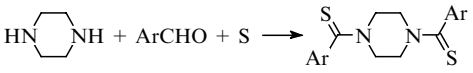
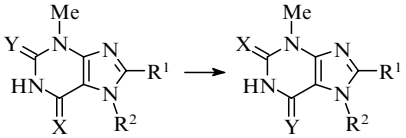
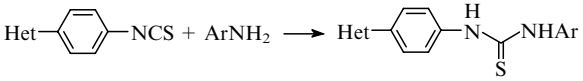
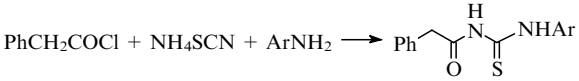
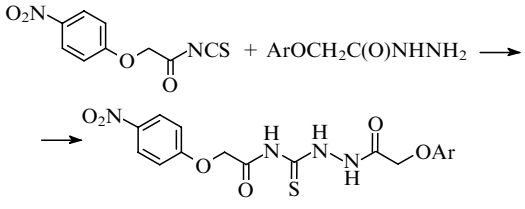
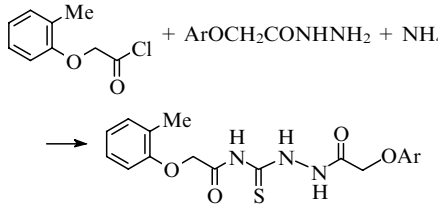
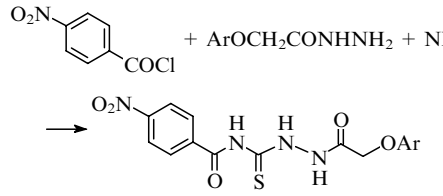
No.	Reaction	Conditions	Number of examples	Yield (%)	Ref.
14	 X = C: R^1-R^6 = H, Alk, Hal, OMe, CO_2Me; X = N: R^1 and R^5 are absent, $R^2 = R^4$ = H, R^3 = Br, R^6 = H, Me.	MW, 8.5 min	28	34–98	195
15	 R = Alk, Ar.	*a*) MW, 15–30 min *b*) Δ, 10 min	14 14	68–93 0–74	196 196
16		$Pd(OAc)_2L$, Bu^tOK, , MW, 15–20 min	9	59–94	197
17	 R = Alk, Ar, Het.	*a*) $NaBO_3 \cdot 4H_2O$, H_2O–EtOH, MW, 2–4 min *b*) MnO_2/SiO_2, MW, 14 min	8 9	83–97 40–99	198 199
18	 R^1, R^2 = Alk, Ar.	*a*) zeolite HY, MW, 2–3 min *b*) zeolite HY, PhMe, N_2, Δ, 9–11 h	8 8	82–95 56–67	200 200
19	 R^1 = Ar, Bn; R^2 = Alk; R^3 = H, Alk.	MW, 8 min	20	38–91	201
20		*a*) DMF, MW, 1.5–10 min *b*) Δ, 5 min	10 1	53–83 48	202 202
21	 R^1, R^2 = H, Me; X = O, Y = S; X = S, Y = O.	MW, 5–10 min	8	90–99	203
22		MW, 2–4.5 min	9	83–95	204
23		PEG-400, $CHCl_3$, MW, 14 min	10	86–94	205
24		MW, 4 min	8	82–95	206

Table 14 (continued).

No.	Reaction	Conditions	Number of examples	Yield (%)	Ref.
25		PEG-400, $CHCl_3$ – DMF, MW, 13 min	12	88 – 98	207
26		PEG-400, $CHCl_3$ – DMF, MW, 13 min	13	82 – 95	208

[a] DIC is *N*,*N*-diisopropylcarbodiimide.

hydroxylamine (No. 18) was also reported. The MWI synthesis of thioanalogues of carboxylic acid amides is illustrated with reactions Nos 19 and 20, whereas the synthesis of thioureas is represented by reactions Nos 21 – 23. 1,4-Disubstituted thiosemicarbazides were obtained by nucleophilic substitution under solvent-free MWI conditions (Nos 24 – 26).

Some of the aforementioned reactions are impossible without microwave activation (Nos 4 and 15), which implies MWE manifestation. The involvement of MWE is also supported by increases in the yields of amines in the MWI-activated reactions as compared to the thermally activated ones (see reactions Nos 3, 4, 7, 18 and 20).

XII. Preparation of nitriles

Aliphatic and aromatic nitriles can be prepared in high yields using the microwave-assisted reaction of aldehydes with hydroxylamine hydrochloride (Table 15, No. 1). The reaction is performed either in the presence of ammonium acetate (conditions *a*) or under solvent-free conditions over zeolites (conditions *b*) or in *N*-methyl-2-pyrrolidone (conditions *d*). Aromatic nitriles can also be synthesised by the dehydration of the respective oximes (No. 2) with phthalic anhydride either in the presence of a solvent (*a*) or using molecular sieve A4 in the absence of solvents (*b*) or on the alumina surface in the presence of tetrachloropyridine (*c*). A catalytic conversion of aryl and vinyl halides into nitriles by the substitution of the cyano group for the bromine atom was realised (No. 3). In all those cases, the use of MW accelerated the reactions but did not noticeably increase the yields of products.

XIII. Ionic liquids

Examples of the microwave-assisted preparation of ionic liquids composed of heterocyclic cations and inorganic anions are compiled in Table 16. For this purpose, the interaction of 1-methylimidazole (Nos 1 and 2) or pyridine (No. 3) with alkyl halides was exploited. The results presented indicate that the reaction times of the MW-activated reactions were substantially shorter than those of the thermal reactions at nearly the same yields of products.

Table 15. Synthesis of nitriles.

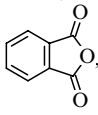
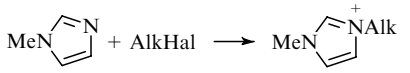
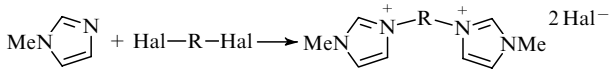
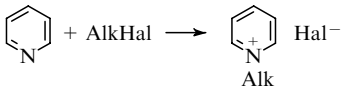
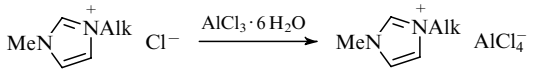
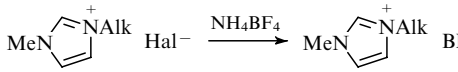
No.	Reaction	Conditions	Number of examples	Yield (%)	Ref.
1	$RCHO \xrightarrow{NH_2OH \cdot HCl} RCN$				
	R = Alk, Ar.	*a*) $AcONH_4$, MW, 1 – 3 min	12	80 – 95	209
		b) zeolite HY, MW, 1 – 2 min	12	84 – 96	200
		c) zeolite HY, PhMe, N_2, Δ, 6 – 8 h	12	68 – 79	200
		d) NMP, MW, 2 – 5 min	8	47 – 96	210
2	ArHC=N–OH ⟶ ArCN	*a*) , MeCN, MW, 3 – 15 min	10	78 – 94	211
		b) molecular sieves A4, MW, 4 – 10 min	14	30 – 95	212
		c) Al_2O_3, tetrachloropyridine, MW, 35 – 180 min	15	60 – 85	213
3	RBr + $Zn(CN)_2$ ⟶ RCN	$Pd(PPh_3)_4$, DMF			214
	R = Ar, $R^1R^2C=CH$.	*a*) MW, 2 – 2.5 min	8	78 – 95	
		b) Δ, 2 – 16 h	7	71 – 97	

Table 16. Synthesis of 1,3-dialkylimidazolium and 1-alkylpyridinium salts.

No.	Reaction	Conditions	Number of examples	Yield (%)	Ref.
1		*a*) MW, 75 – 240 s	7	82 – 100	215
		b) MW, 1 – 2.5 min	10	71 – 94	216
		c) Δ, 3 – 5 h	10	50 – 95	216
2		*a*) MW, 50 – 90 s	8	78 – 94	216
		b) Δ, 3 – 5 h	8	56 – 97	216
3		MW, 400 – 600 s	2	100	215
4		MW, 2 – 4 min	8	100	109
5		*a*) MW, 2.5 – 3.5 min	9	88 – 92	217
		b) Δ, 3 h	3	28 – 36	217

MWI also enabled anion exchange reactions (Nos 4, 5) significantly increasing their yields as compared to the thermal conditions. This means that the MWE is involved.

XIV. Synthesis of heterocyclic compounds

1. Three-membered heterocycles

Chanda *et al.*[218] described a method for the catalytic microwave-assisted synthesis of diverse aziridines by the reaction of olefins with sodium haloamines (TsNXNa) (Table 17, No. 1). The oxidation of alkenes with hydrogen peroxides under MWI afforded epoxides (No. 2), whereas the reductive cyclisation of α-haloketones and diethyl dithiophosphoric acid in the presence of alumina-supported sodium borohydride yielded thiiranes (No. 3). The yields of products depend on the structure of reagents and the reaction type. The MWE was manifested in all those cases both in the significant acceleration of the reactions and increases of yields of products.

2. Four-membered heterocycles

Table 18 lists some examples of the microwave-assisted preparation of four-membered heterocyclic compounds (*e.g.*, β-lactams, oxetanes and thietanes). The Staudinger reaction was utilised for the synthesis of β-lactams (Table 18, Nos 1 – 5). Oxetanes were prepared by the [2 + 2]-cycloaddition of alkynes to β-formylenamines. The reaction was accomplished over the alumina surface under solvent-free conditions (No. 6). Functionalised thietanes were prepared using the nucleophilic addition of $HSP(S)(OEt)_2$ to the double bond of α,β-unsaturated ketones followed by the reductive cyclisation initiated by nucleophiles (No. 7).

3. Five-membered heterocycles

a. Five-membered heterocycles with one nitrogen atom

The microwave-assisted three-component condensation of amines, ketones (or aldehydes) and nitro compounds was utilised to synthesise substituted pyrroles (Table 19, Nos 1 – 3). Functionalised indoles were prepared by the Fischer reaction (No. 4) and by the condensation of *o*-iodoanilines with alkynes (Nos 5 and 6).

Table 17. Synthesis of three-membered heterocyclic compounds.

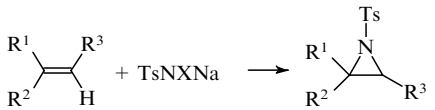
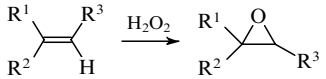
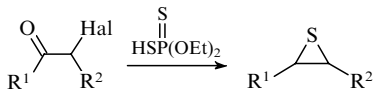
No.	Reaction	Conditions	Number of examples	Yield (%)	Ref.
1		$CuCl_2$, MW, 12 min	5	36 – 88	218
	R^1 = Alk, Ar; R^2 = H, Alk; R^3 = H, Alk, CO_2Me; X = Cl, Br.				
2		*a*) hydrotalcite, MeCN, MW, 1 – 8 min	13	62 – 100	219
		b) Δ, 4 – 24 h	13	25 – 100	219
	R^1 = Alk; R^2, R^3 = H, Alk.				
3		*a*) $NaBH_4 - Al_2O_3$, MW, 2 – 4 min	11	81 – 94	220
		b) Δ, 11 – 15 h	11	39 – 49	220
	R^1 = Alk, Ar; R^2 = H, Alk, Ar.				

Table 18. Microwave-assisted preparation of four-membered heterocyclic compounds with nitrogen, oxygen or sulfur atoms.

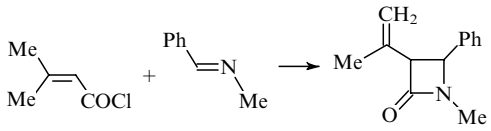
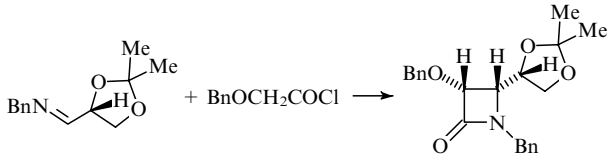
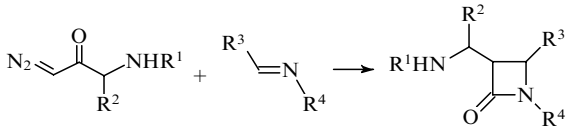
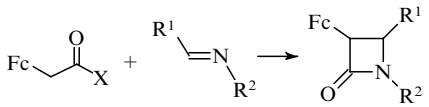
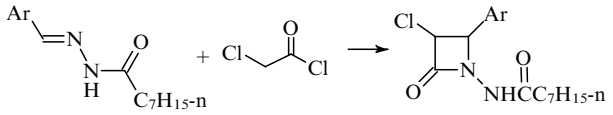
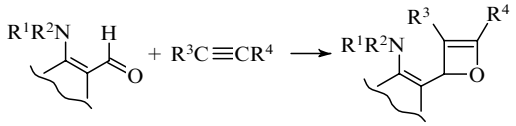
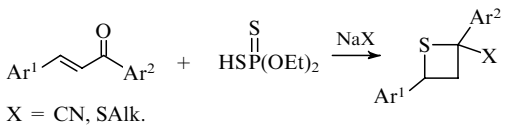
No.	Reaction	Conditions	Number of examples	Yield (%)	Ref.
1		PhCl, Et_3N, MW, 0.5 – 10 min	8	70 – 75	221
2		CH_2Cl_2, Et_3N, MW, 3 min	1	90	222
3	 R^1 = Cbz, Boc, Fmoc; R^2 – R^4 = Alk.	*o*-$Cl_2C_6H_4$, MW, 5 min	7	40 – 85	223
4	 R^1, R^2 = Alk, Ar; X = Cl, OH.	*a*) $PhOP(O)Cl_2$, Et_3N, PhMe, MW, 4 – 6 min	10	17 – 96	224
		b) Δ, 3.5 – 6 h	15	17 – 58	224
5		*a*) Et_3N, CH_2Cl_2, MW, 5 – 8 min	5	70 – 78	225
		b) Δ, 5 – 8 h	5	55 – 62	225
6	 R^1, R^2 = H, Alk; R^3, R^4 = H, Alk, Ar, CO_2Et.	Al_2O_3, MW, 5 – 8 min	6	72 – 80	226
7	 X = CN, SAlk.	Al_2O_3, MW, 2 – 4 min	9	77 – 92	227

Table 19. Synthesis of pyrrole derivatives.

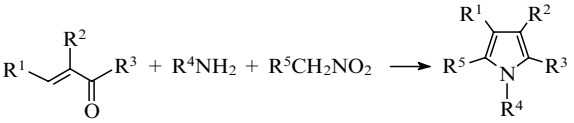
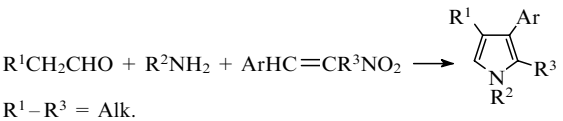
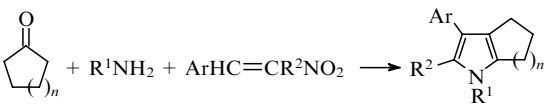
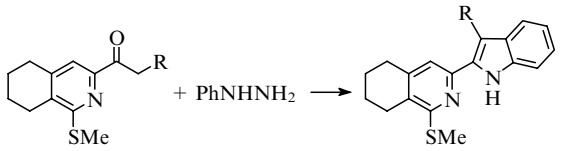
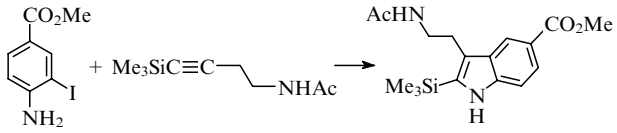
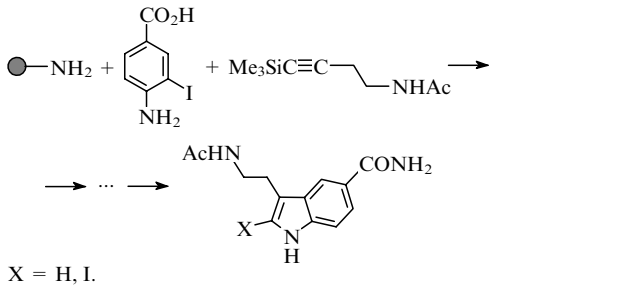
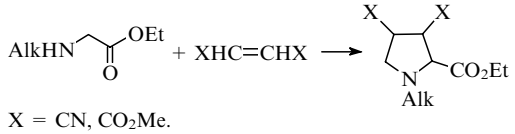
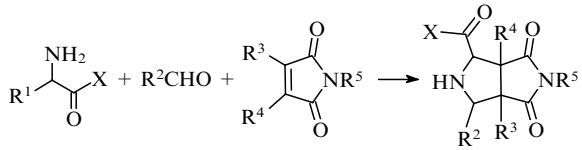
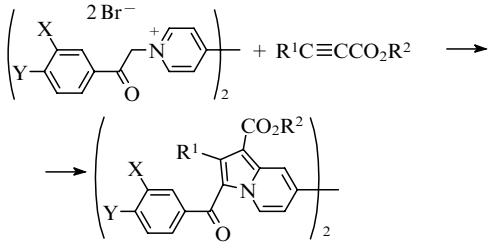
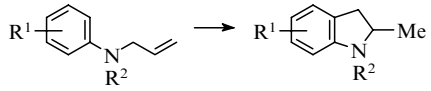
No.	Reaction	Conditions	Number of examples	Yield (%)	Ref.
1	 R^1 = H, Alk, Ar; R^2, R^5 = H, Me, Ph; R^3, R^4 = Alk, Ar.	SiO_2, MW, 5 – 10 min	19	60 – 72	228
2	 R^1 – R^3 = Alk.	Al_2O_3, MW, 13 – 15 min	12	71 – 81	228
3	 R^1, R^2 = Alk; n = 1 – 4.	Al_2O_3, MW, 13 – 15 min	15	74 – 86	228

Table 19 (continued).

No.	Reaction	Conditions	Number of examples	Yield (%)	Ref.
4	 R = H, Et.	*a*) K-10, $ZnCl_2$, MW, 35–180 min *b*) Δ, 1-MeNaph, 3 h	2 2	50–65 25–35	229 229
5		$Pd(OAc)_2$, AcONa, Ph_3P, LiCl, DMA [a] or DMF–MeCN, MW, 8–17 min (DMF–MeCN) or 30–120 min (DMA)	9 [b]	72–84 (DMF–MeCN) 34–63 (DMA)	230
6	 X = H, I.	*a*) MW, 3–26 min *b*) Δ, 4–48 h	6 6	8–100 56–100	231 231
7	 X = CN, CO_2Me.	MW, 15 min	6	35–78	232
8	 R^1, R^3, R^4 = H, Alk; R^2, R^5 = Alk, Ar; X = OH, OMe.	$C_2H_4Cl_2$, MW, 5 min	10	0–89	233
9	 R^1 = H, CO_2Et; R^2 = Me, Et; X, Y = H, Hal, NO_2, OMe.	*a*) KF/Al_2O_3, MW, 7–10 min *b*) Δ, 3–6 h	8 8	81–93 50–59	234 234
10	 R^1 = H, Me, F, Cl, NO_2, OMe, CN; R^2 = H, Me, Et, Bn.	Zn^{2+}-montmorillonite, MW, 6–9 min	10	70–85	235

[a] DMA is *N*,*N*-dimethylacetamide; [b] different temperature modes and solvents were tested.

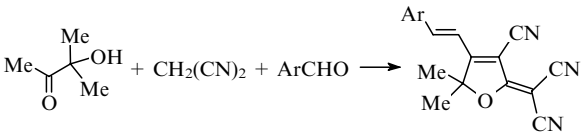
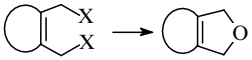
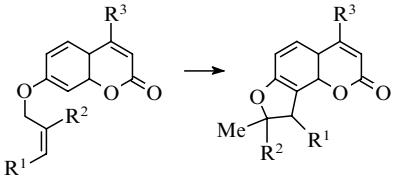
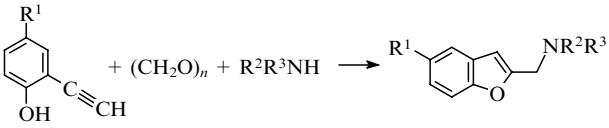
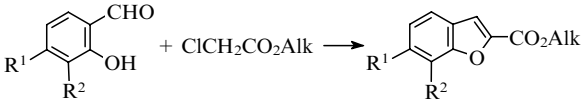
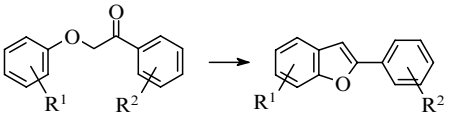
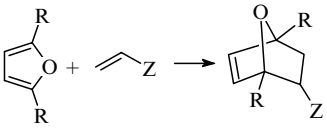
The coupling of α-amino acids with alkenes afforded pyrrolidine derivatives (Nos 7 and 8). The microwave 1,3-dipolar cycloaddition of alkynes to pyridinium salts was applied to the synthesis of 7,7′-bisindolizines (No. 9). Indolines were synthesised by the Cope rearrangement from *N*-allylamines (No. 10).

In the case of the Fischer synthesis of indoles (No. 4), the reaction proceeded at the same rates under microwave and thermal activations, but the yields of products were 2 times higher in the former mode. An increase in the yield of products under microwave irradiation (by 30%, on average) was also registered for the 1,3-dipolar cycloaddition (No. 9), which is indicative of the MWE.

b. Five-membered heterocycles with one oxygen atom

The three-component condensation of α-hydroxy diketones with malonic dinitriles and aldehydes (Table 20, No. 1), intramolecu-

Table 20. Synthesis of furan derivatives.

No.	Reaction	Conditions	Number of examples	Yield (%)	Ref.
1		EtONa – EtOH, MW, 30 min	8	65 – 84	236
2	 X = Ar; X = Cl, Br.	*a*) Al_2O_3, MW, 5 – 15 min *b*) Al_2O_3, C_6H_{14}, Δ, 20 – 90 min	5 5	59 – 82 49 – 98	237 237
3	 R^1 = H, Me, Ph; R^2, R^3 = H, Me.	$BF_3 \cdot Et_2O$, NMF,[a] MW, 18 – 25 min	11	54 – 74	238
4	 R^1 = H, Me, C(O)Me; R^2 = Alk, Ph; R^3 = Alk.	CuI – Al_2O_3, MW, 5 min	12	38 – 70	239
5	 R^1, R^2 = H, Alk, NEt_2, OMe.	K_2CO_3, Bu^n_4NBr, MW, 8 – 10 min	12	65 – 96	240
6	 R^1 = H, Hal, Alk; R^2 = Me, Ph.	KSF, MW, 2 – 6 min	14	76 – 92	241
7	 R = H, Me; Z = CN, CO_2Me.	*a*) SiO_2 or Si(M) (M = Ti, Al, Zn), MW, 7 – 45 min *b*) 20 °C, 2 – 24 h	11 14	0 – 80 0 – 79	242 242

[a] NMF is *N*-methylformamide.

lar cyclisation of dihalides (No. 2), rearrangement of phenol and allyl alcohol ethers (No. 3) were applied to the microwave synthesis of dihydrofurans. Benzofurans were prepared by the Mannich reaction (No. 4), condensation of chloroacetic acid esters with substituted salicylic aldehydes under phase-transfer catalysis conditions (No. 5) and intramolecular cyclisation of α-phenoxyacetophenones (No. 6). The condensation of furans (or alkylfurans) with acrylic acid derivatives giving rise to tetrahydrofurans (No. 7) was performed under the conditions of both microwave and thermal activation. The microwave reaction was 32 times faster than the reaction activated by convection heating at nearly the same yields of products. The use of MWI also accelerated the synthesis of dihydrofurans (No. 2) but the yields of products were sometimes lower as compared to the thermally activated reactions.

c. Five-membered heterocycles with two nitrogen atoms

The microwave-assisted synthesis of pyrazole derivatives from hydrazines and compounds with carbonyl groups was realised (Table 21, Nos 1 – 5). Non-substituted pyrazolylpyrazolino[60]-fullerenes were synthesised by the 1,3-dipolar cycloaddition of C_{60} to arylhydrazones (No. 6), whereas the *trans*-amination of dimethylaminoacrylates followed by the cyclo-condensation of enehydrazines afforded 3-pyrazolones (No. 7). Non-substituted triarylimidazoles can be prepared by the condensation of α-diketones, aldehydes and ammonium acetate; the condensation of the same reagents with amines afforded substituted derivatives (No. 8). Indolyl-substituted imidazoles were obtained by cyclisation of the respective ketoamides with ammonium acetate (No. 9). Benzimidazole derivatives were synthesised by the microwave cyclocondensation of *N*-trifluoromethylacetylphenylenediamine (No. 10) and condensation of *o*-phenylenediamines with carbonyl compounds (Nos 11 – 13). Imidazolones can be prepared by the

Table 21. Synthesis of pyrazole and imidazole derivatives.

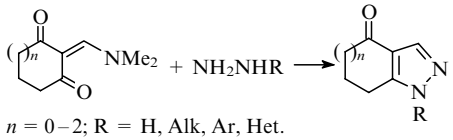
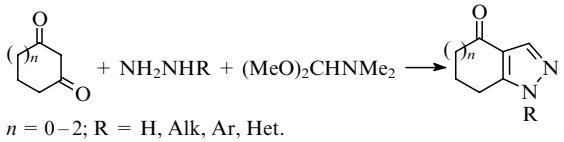
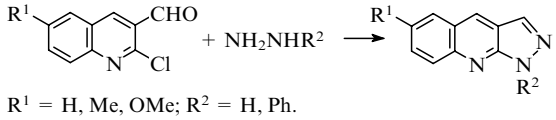
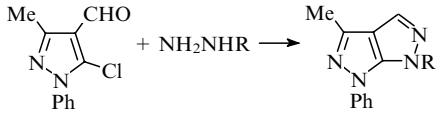
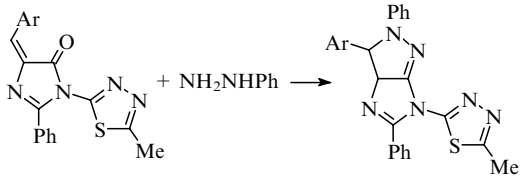
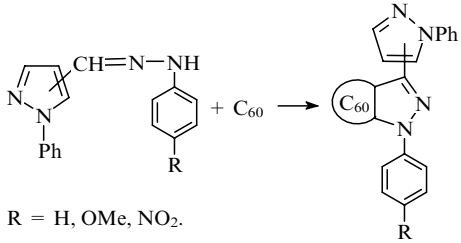
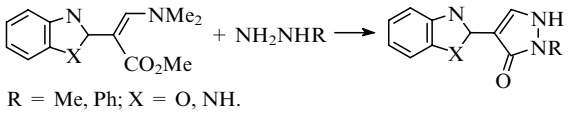
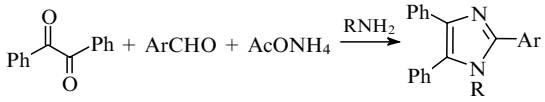
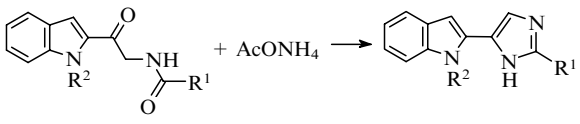
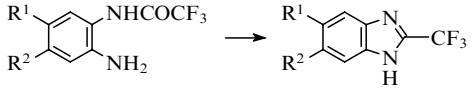
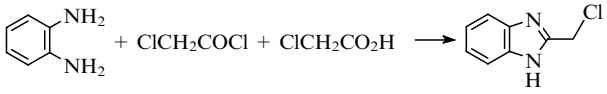
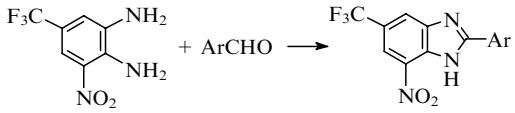
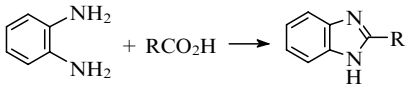
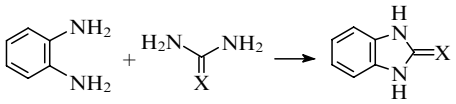
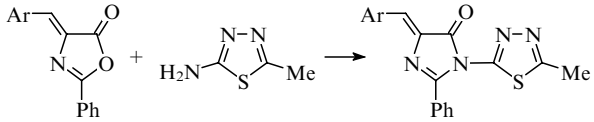
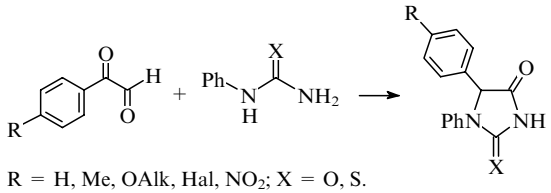
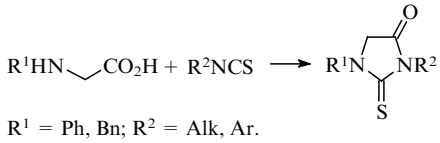
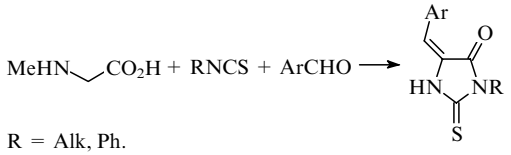
No.	Reaction	Conditions	Number of examples	Yield (%)	Ref.
1	 n = 0–2; R = H, Alk, Ar, Het.	AcOH, H_2O, MW, 2 min	9	65–99	243
2	 n = 0–2; R = H, Alk, Ar, Het.	AcOH, H_2O, MW, 2 min	6	66–87	243
3	 R^1 = H, Me, OMe; R^2 = H, Ph.	TsOH, MW, 1.5–2 min	6	78–97	244
4	 R = H, Ph.	TsOH, MW, 2–2.5 min	2	94–96	244
5		*a*) Al_2O_3, MW, 5–6 min *b*) Δ, 7.5–8 h	3 3	89–92 70–74	245 245
6	 R = H, OMe, NO_2.	NBS,[a] Et_3N, benzene, MW, 30 min	6	22–44	246
7	 R = Me, Ph; X = O, NH.	*a*) MW, 30 min *b*) Δ, 2–264 h	1 2	96 74–92	161 161
8	 R = H, Alk, Ar.	R = H: *a*) zeolite HY, MW, 6 min *b*) SiO_2, MW, 6 min R = Alk, Ar: *a*) zeolite HY, MW, 6 min *b*) SiO_2, MW, 6 min	 8 8 17 17	 80–94 54–89 42–85 60–91	247 248
9	 R^1 = Alk, Ar; R^2 = H, Bn.	*a*) DMF, MW, 10–30 min *b*) DMF, 180 °C, 12–16 h	10 9	50–75 25–75	249 249
10	 R^1, R^2 = H, Me, Cl, NO_2, CO_2Et.	*a*) K-10, MW, 2 min *b*) Δ, 20 h	7 6	84–95 18–31	250 250

Table 21 (continued).

No.	Reaction	Conditions	Number of examples	Yield (%)	Ref.
11		*a*) SiO_2, MW, 4 min *b*) HCl, Δ, 8 h	1 1	90 92	251 251
12		*a*) $ZnCl_2$, Al_2O_3, MW, 4 min *b*) Δ, 2 h	10 10	72–90 60–80	252 252
13	 R = H, Alk, Ar.	PPA,[b] MW, 6–8 min	12	39–88	253
14	 X = O, S.	PPA,[b] MW, 8 min	2	71–86	253
15		*a*) Al_2O_3, MW, 5–6 min *b*) Δ, 7–8 h	3 3	90–94 70–72	245 245
16	 R = H, Me, OAlk, Hal, NO_2; X = O, S.	PPE,[c] MW, 2.5–3.5 min	16	80–95	254
17	 R^1 = Ph, Bn; R^2 = Alk, Ar.	*a*) $Et_3N-C_2H_4Cl_2$, MW, 5 min *b*) PS–DMAP,[d] MeCN, MW, 5 min	5 4	57–91 56–87	255 256
18	 R = Alk, Ph.	*a*) MW, 12 min *b*) Δ, 12 h	56 56	69–94 65–98	256 256

Hereinafter, [a] NBS is *N*-bromosuccinimide; [b] PPA is polyphosphoric acid; [c] PPE is polyphosphoric ester; [d] PS–DMAP is polystyrene-grafted dimethylaminopyridine.

reactions of *o*-phenylenediamine with urea or thiourea (No. 14) or of hetarylamines with oxazolones (No. 15). The condensation of α-ketaldehydes with urea or thiourea can be applied to the preparation of hydantoins and thiohydantoins (No. 16); the latter compounds were also synthesised by the microwave cyclisation of glycine with isothiocyanates (Nos 17 and 18).

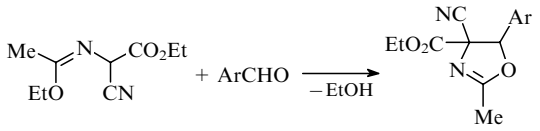
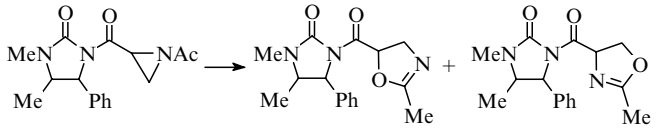
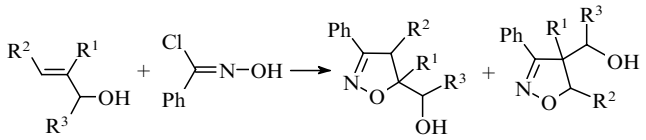
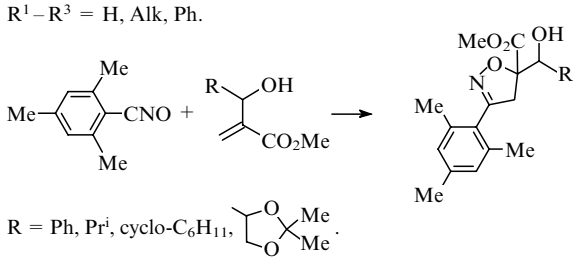
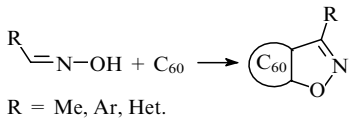
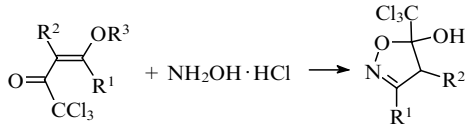
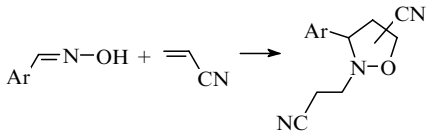
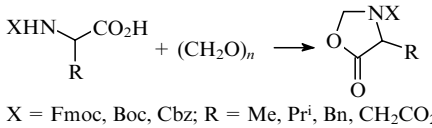
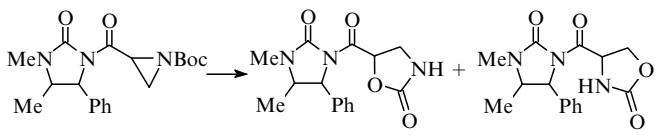
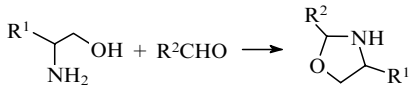
The yields of reaction products varied over a wide range, depending on the structure of reagents. In the majority of cases, the use of MWI accelerated the reactions.

d. Five-membered heterocycles with one nitrogen and one oxygen atom

Two methods, *viz*., the 1,3-dipolar cycloaddition of aromatic aldehydes to imidates, *i.e*., potential azomethine ylides, (Table 22, No. 1) and the intramolecular rearrangement of the aziridine ring (No. 2) were used in the microwave synthesis of oxazolines.

Isoxazolines were synthesised by the 1,3-dipolar cycloaddition of oximes to allyl alcohols (Table 22, Nos 3–5) or of α,β-unsaturated ketones to hydroxylamine (No. 6) under microwave activation. Isoxazolidines were prepared in a similar way using the 1,3-dipolar cycloaddition of alkenes to oximes (No. 7).The condensation of α-amino acid derivatives with formaldehyde (No. 8) or other aldehydes (see Table 11, No. 15) and the intramolecular rearrangement of the aziridine ring (Table 22, No. 9) were utilised in the synthesis of oxazolones, whereas oxazolidines were prepared similarly from α-aminoalcohols and aldehydes (No. 10). Although the use of MWI accelerated the reactions in some cases as compared to the thermal activation, the yields of products either remained unchanged or even decreased.

Table 22. Microwave-assisted synthesis of oxazole and isoxazole derivatives.

No.	Reaction	Conditions	Number of examples	Yield (%)	Ref.
1		*a*) MW, 1–4.5 h *b*) Δ, 1–6 h	6 6	87–98 86–96	257 257
2		$M(OTf)_2$ (M = Zn, Cu) or $BF_3 \cdot Et_2O$, or $MgBr_2 \cdot Et_2O$; THF or PhMe, MW, 10 min	7	50–95[a]	258
3	 R^1–R^3 = H, Alk, Ph.	*a*) Et_3N, benzene, MW, 30–120 s *b*) Δ, 50 h	22 11	15–90[a] 40–84[a]	259 259
4	 R = Ph, Pr^i, cyclo-C_6H_{11},	*a*) MeMgBr, PhCl or CCl_4, MW, 1.5–90 min *b*) CH_2Cl_2, 20 °C, 24–78 h *c*) PhMe, 80 °C, 2–4 h	4 6 5	34–99 35–96 56–99	260 260 260
5	 R = Me, Ar, Het.	NBS, Et_3N, MW, 30 min	8	16–36	261
6	 R^1 = H, Alk, Ar; R^2, R^3 = H, Alk.	*a*) PhMe–Py, MW, 6 min *b*) HCl–MeOH, Δ, 16 h	13 13	78–95 60–90	262 262
7		*a*) MW, 9–30 min *b*) Δ, 12–120 h *c*) *hv*, 72–168 h	6 6 5	73–93[a] 71–98[a] 66–81[a]	263 263 263
8	 X = Fmoc, Boc, Cbz; R = Me, Pr^i, Bn, CH_2CO_2H ù óp.	TsOH, PhMe, MW, 3–6 min	14	81–98	264
9		$M(OTf)_2$ (M = Zn, Cu) or $BF_3 \cdot Et_2O$, PhMe, MW, 10 min	4	65–99[a]	265
10	 R^1 = Bu^t, Ph, Bn; R^2 = Bu^t, *p*-FC_6H_4.	MW, 1–3 min	14	55–98	266

[a] The total yield of isomers is given.

The synthesis of isoxazolidines (No. 7) is worth a special discussion. Kusurkar and Kannadkar[263] compared three types of activation, *viz*., UV, thermal and microwave. The reaction time under the microwawe conditions was shorter than 0.5 h, whereas it was in the range of 12–120 h and 72–168 h for the thermal and

UV activations, respectively. Furthermore, the UV activation provided the minimum yields of products.

e. Five-membered rings with three or more heteroatoms

1,2,3-Triazoles were synthesised by the microwave-assisted condensation of organic azides with triphenylacetylmethylenephosphoranes (Table 23, No. 1) or acetylenecarboxylic amide (No. 2). The condensation of aromatic aldehydes with hydrazine, hydrazine hydrochloride or hydroxylamine hydrochloride under MWI yielded 1,2,4-triazoles (No. 3), whereas 1,2,4-triazolines were produced by the three-component condensation of isatin with anilines or thiosemicarbazide (No. 4). The intramolecular cyclisation of hydrazine, semicarbazide and thiosemicarbazide derivatives afforded oxadiazoles and thiadiazoles (Nos 5–9). The reaction of aryl cyanides with sodium azide gave rise to tetrazoles (No. 10). For all these reactions, the yields of products depended on the nature of substituents in reagents.

4. Six-membered heterocycles

a. Six-membered heterocycles with one nitrogen atom

Substituted pyridines can be synthesised using the Bohlmann–Rahtz (Table 24, No. 1) or Diels–Alder (Nos 2 and 3) reactions or by the aromatisation of 1,4-dihydropyridine with various oxidants (No. 4) (urea nitrate was found to be the best one). Symmetrical dipyrazolopyridine was afforded by the condensation of 5-amino-1,3-dimethylpyrazole with *p*-methylbenzaldehyde (No. 5). The three-component condensation of aldehydes, secondary amines and 2-azidobenzophenones gave rise to quinoline derivatives (No. 6) and the condensation of 2-amino substituted benzaldehyde or benzimides with different heterocyclic compounds yielded polycondensed quinolines (No. 7). The condensation of diarylamine with carboxylic acids (the Bernthsen reaction) was applied to the synthesis of acridine (Nos 8–10). The Hantzsch method was used to synthesise 1,4-dihydropyridine derivatives (Nos 11–15), and 1,2-dihydroquinolines were prepared by the Skraup reaction (No. 16). Dihydroisoquinolines

Table 23. Synthesis of triazole, oxadiazole, thiadiazole and tetrazole derivatives.

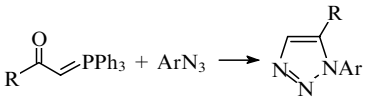
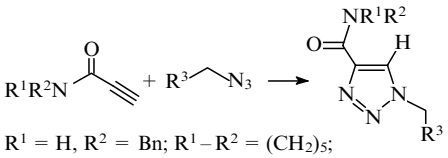
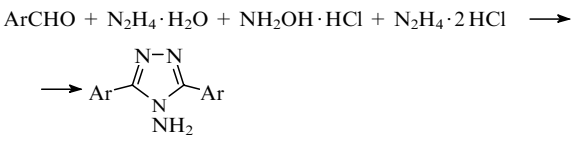
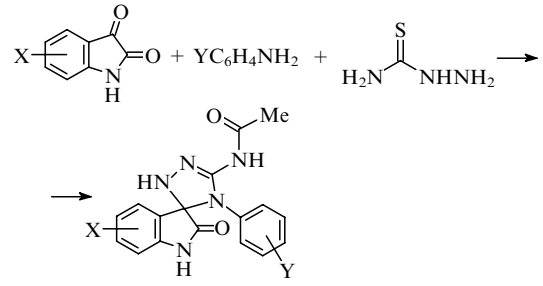
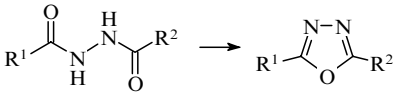
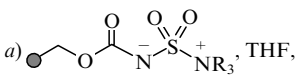
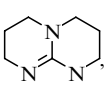
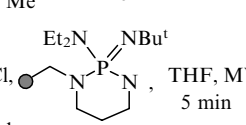
No.	Reaction	Conditions	Number of examples	Yield (%)	Ref.
1	 $R = Alk, Ar.$	SiO_2, MW, 4–10 min	6	67–82	267
2	 $R^1 = H, R^2 = Bn; R^1–R^2 = (CH_2)_5;$ $R^3 = Ph, PhCH_2CH_2, MeCCH_2OCH_2.$	MW, 30 min	6	62–84	268
3		NMP, $(CH_2OH)_2$, MW, 5–12 min	8	42–67	210
4	 $X = H, Hal, NO_2; Y = CF_3.$	AcOH, MW, 4–9 min	5	80–86	269
5	 $R^1 = H, Alk, NHX (X = Alk', Bn), Ar; R^2 = Me, Ph.$	*a*) , THF, , Amberlyst 15, MW, 5 min	16	0–100	270
		b) TsCl, , THF, MW, 5 min	16	49–100	270
		c) Δ, 4 h	16	38–100	270

Table 23 (continued).

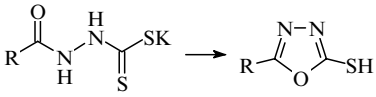
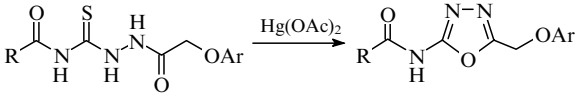
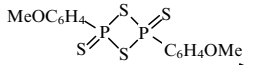
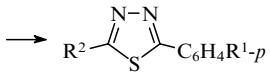
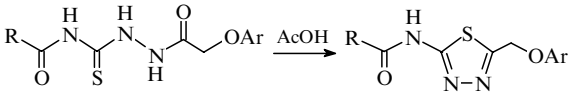
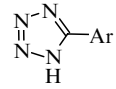
No.	Reaction	Conditions	Number of examples	Yield (%)	Ref.
6	 R = Ar, Bn.	DMF or DMSO, or Py, MW, 0.5–2 min	27	69–84	271
7		*a*) R = *p*-ClC_6H_4: AcOH, MW, 4.5 min	10	86–94	272
		b) R = *p*-$MeOC_6H_4OCH_2$: AcOH, MW, 4.5 min	8	86–91	273
8	*p*-$R^1C_6H_4C(O)NHNHC(O)R^2$   R^1 = H, Br, OMe; R^2 = Alk, Ar, OAlk, OAr.	MW, 3–13 min	15	0–95	274
9	 R =  (*a*, *b*), *o*-ClC_6H_4  (*c*), *o*-$MeC_6H_4OCH_2$ (*d*).	*a*) MW, 5 min	10	94–98	275
		b) Δ, 6 h	3	78–85	275
		c) MW, 5 min	10	86–98	276
		d) MW, 5 min	10	91–97	277
10	ArCN + NaN_3 	NH_4Cl, DMF	8	36–98	214
		a) MW, 10–25 min			
		b) Δ, 3–96 h	6	35–97	214

Table 24. Synthesis of pyridine derivatives.

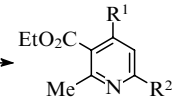
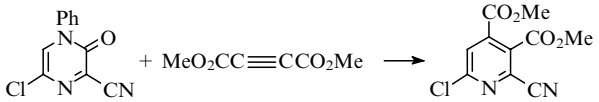
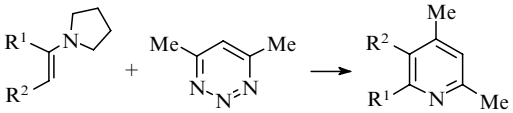
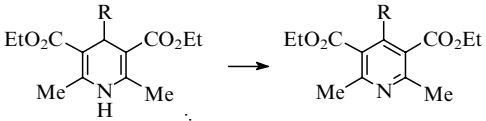
No.	Reaction	Conditions	Number of examples	Yield (%)	Ref.
1	$NH_2C(Me)=CHCO_2Et$ + $R^1C\equiv CC(O)R^2$  R^1 = H, Me, Ph, $SiMe_3$; R^2 = Me, Ar.	*a*) DMSO, MW, 10–20 min	5	24–94	278
		b) DMSO, Δ, 10 min	1	27	278
2		$C_2H_4Cl_2$, $(bmim)PF_6$, MW, 5 min	1	82	279
3	 R^1, R^2 = Alk.	MW, 3–20 min	11	18–71	280
4	 R = H, Alk, Ar.	*a*) $H_2N(C=O)NHONO_2$, MeCN, MW, 0.5–2 min	14	89–98	281
		b) $K_2S_2O_8$/Co(II), MeCN, MW, 10–15 min	14	80–94	281
		c) $K_2S_2O_8$/Co(II), MeCN, Δ, 1.5–3 h	14	18–96	281

Table 24 (continued).

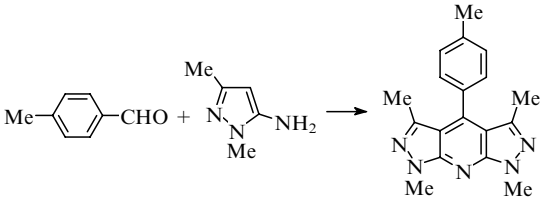
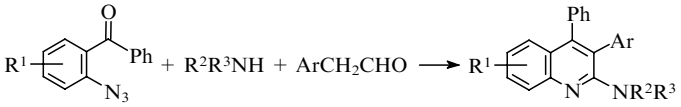
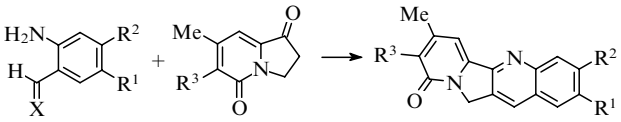
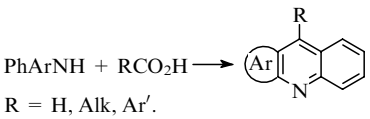
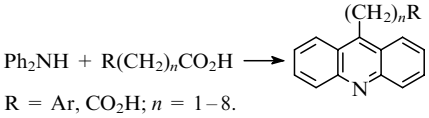
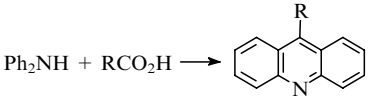
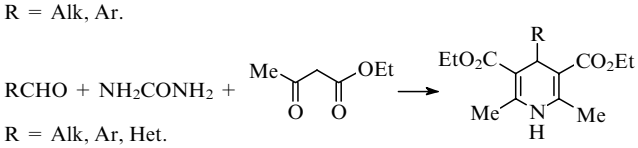
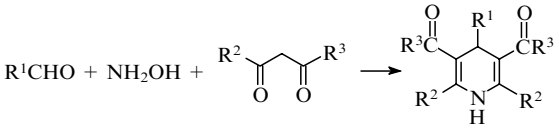
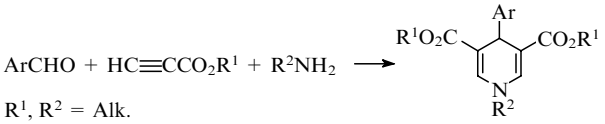
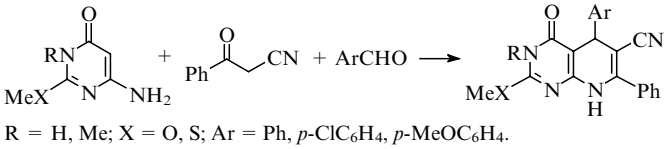
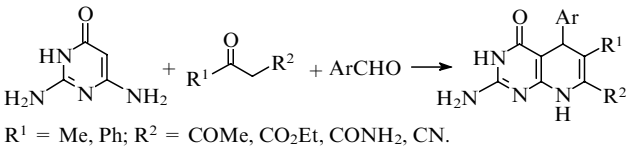
No.	Reaction	Conditions	Number of examples	Yield (%)	Ref.
5		MW, 15 min	1	91	282
6	 R^1 = H, Me, Hal, NO_2; R^2, R^3 = Alk, Ar.	$C_2H_4Cl_2$, MW, 10 min	7	57–100	283
7	 X = O, NAr; R^1, R^2 = H, OAlk; R^3 = CN, $CONH_2$, Alk.	DMF, Et_2O or EtOH, AcOH *a*) MW, 15 min *b*) Δ, 8 h	 9 9	 57–91 31–51	284
8	 R = H, Alk, Ar′.	$ZnCl_2$, MW, 5–22 min	16	0–87	285
9	 R = Ar, CO_2H; n = 1–8.	$ZnCl_2$, MW, 4–8 min	9	50–80	286
10	 R = Alk, Ar.	*a*) $ZnCl_2$, MW, 2.5–12 min *b*) $ZnCl_2$, Δ, 2.5–12 min	7 7	57–98 18–53	287 287
11	 R = Alk, Ar, Het.	*a*) SiO_2, MW, 2–6 min *b*) Δ, 12 h	14 13	70–93 15–77	288 288
12	 R^1 = Alk, Ar; R^2 = Me: R^3 = OEt, OBu^t, OBn; R^2–R^3 = $CH_2CMe_2CH_2$.	*a*) MW, 4–10 min *b*) Δ, 12 h	28 8	21–89 15–72	289 289
13	 R^1, R^2 = Alk.	SiO_2, MW, 4 min	8	62–94	290
14	 R = H, Me; X = O, S; Ar = Ph, *p*-ClC_6H_4, *p*-$MeOC_6H_4$.	*a*) MW, 15–20 min *b*) EtOH, Δ, 40–48 h	7 7	70–75 21–25	291 291
15	 R^1 = Me, Ph; R^2 = COMe, CO_2Et, $CONH_2$, CN.	*a*) DMSO, $ZnBr_2$, MW, 20 min *b*) Δ, 3 days	10 10	55–91 14–86	292 292

Table 24 (continued).

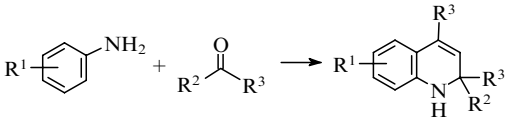
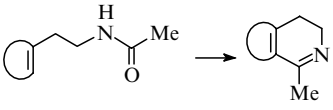
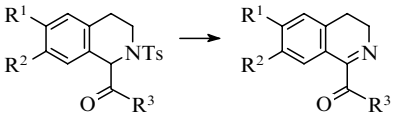
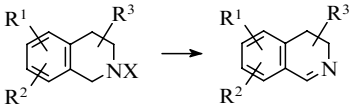
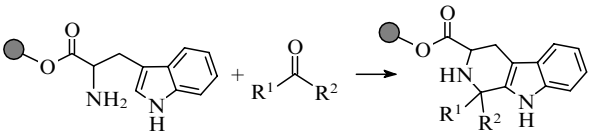
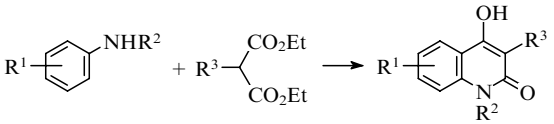
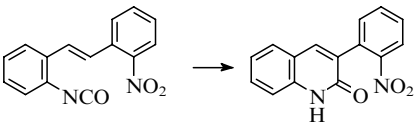
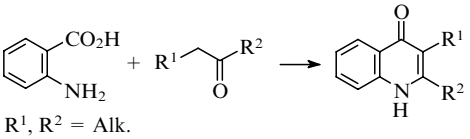
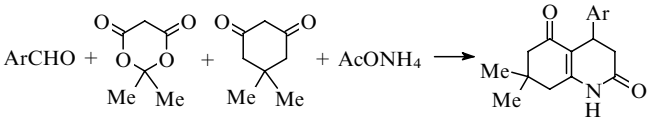
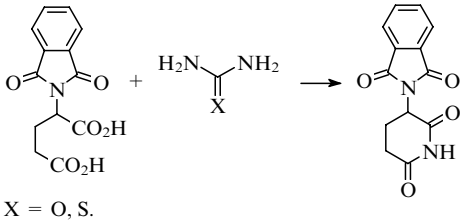
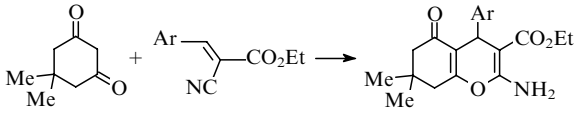
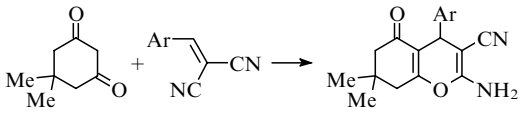
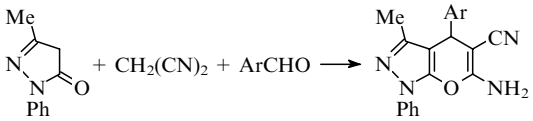
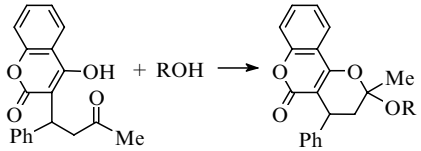
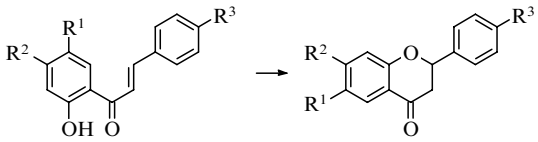
No.	Reaction	Conditions	Number of examples	Yield (%)	Ref.
16	 R^1 = H, Alk, Ar, Het, Hal, OMe; R^2 = Alk; R^3 = Alk, Ar.	$Sc(OTf)_3$, MeCN, MW, 50–60 min	44	30–98	293
17	 =  (**A**),  (**B**).	*a*) $POCl_3$, P_2O_5, MeCN (**A**) or PhMe (**B**), MW, 600 s (**A**) or 160 s (**B**)	2	75 (**A**), 86 (**B**)	294
		b) MeCN, Δ, 2 h (**B**)	1	37 (**B**)	294
18	 R^1, R^2 = H, OAlk; R^3 = Alk, Ar.	KF/Al_2O_3, MW, 10–20 s	5	60–92	295
19	 R^1, R^2 = H, OMe, OBn: R^3 = Alk, Ar; X = Ts, SPh, COAr.	KF/Al_2O_3, MW, 20–420 s	12	16–86	295
20	 R^1 = Alk, Ar; R^2 = H, Alk, Ar.	TsOH, $CHCl_3$, MW, 15 min	15	87–99	296
21	 R^1 = H, Hal, CF_3, OMe; R^2 = H, Alk; R^3 = Ph, OAlk.	MW, 15 min	19	0–94	297
22		$PhNO_2$, MW, 12 min	1	80	298
23	 R^1, R^2 = Alk.	DMA, MW, 6–10 min	7	56–81	299
24		MW, 3–5 min	7	72–86	300
25	 X = O, S.	MW, 15 min	2	65–85	301

Table 25. Synthesis of six-membered heterocyclic compounds with one oxygen atom.

No.	Reaction	Conditions	Number of examples	Yield (%)	Ref.
1		$AcONH_4$, MW, 4–8 min	7	61–90	302
2		$AcONH_4$, MW, 1.5–4 min	7	89–96	303
3		Py–EtOH, MW, 2–8 min	6	61–91	304
4	 R = Me, Et.	K-10, MW, 2–5 min	4	65–90	305
5	 R^1 = H, Me, Cl; R^2, R^3 = H, OMe.	DBU, Na_2SO_4, MW, 20 min	5	0–27	306

were prepared by the cyclocondensation of the respective amides (No. 17) and direct oxidation of tetrahydroisoquinolines (Nos 18 and 19). The microwave-assisted cyclocondensation of amines (for instance, tryptophan derivatives) with compounds having a carbonyl group (No. 20) was achieved. Table 24 also gives some examples of syntheses of different quinolinones (Nos 21–23), octahydroquinoline-2,5-diones (No. 24) and hexahydropyridine-2,6-diones (No. 25).

Typically, the use of MWI substantially reduced the reaction times as compared to the thermal activation (Nos 7, 14 and 17); in some cases, an increase in the yields of heterocyclic products was registered (see, for instance, Nos 7, 10, 14, and 17), which indicated the MWE.

b. Six-membered heterocycles with one oxygen atom

Table 25 lists examples of applications of MWI to the synthesis of six-membered heterocyclic compounds with one oxygen atom, *viz*., substituted γ-pyrans through the condensation of Meldrum's acid with β-carboxy-β-cyanostyrenes (No. 1) or benzylidenemalonodinitriles (No. 2) or three-component condensation of malonodinitrile, aromatic aldehydes and 3-methyl-1-phenyl-2-pyrazolin-5-one (No. 3), dihydropyrans through the intramolecular cyclisation of hydroxyketones (No. 4) and flavones through the intramolecular cyclocondensation of halkones (No. 5).

For all of the above examples except for No. 5, the reaction times under the MWI conditions were 1.5–8 min and the yields of products were up to 90%, which confirms the high efficiency of this activation method.

c. Six-membered heterocycles with two heteroatoms

The microwave activation appeared equally efficient in the synthesis of heterocyclic compounds with two nitrogen atoms in a ring, such as pyrazine, pyridazine, pyrimidine (Table 26, Nos 1–11) and pyrimidopyrimidine (Nos 12–14). 1,3-Oxazin-2-

Table 26. Synthesis of six-membered heterocyclic compounds with two heteroatoms.

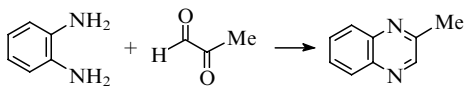
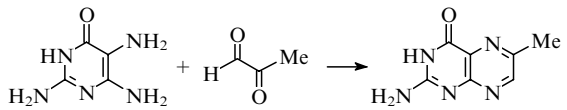
No.	Reaction	Conditions	Number of examples	Yield (%)	Ref.
1		MW, 35–270 s	5	42–95	307
2		MW, 62–270 s	5	28–70	307

Table 26 (continued).

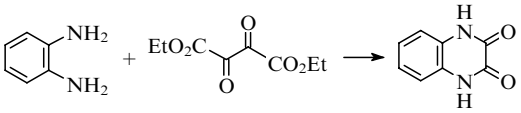
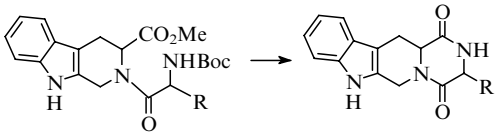
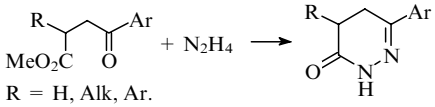
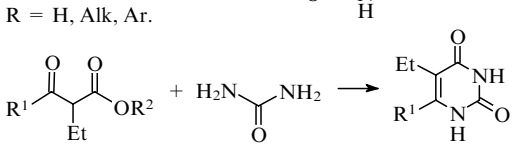
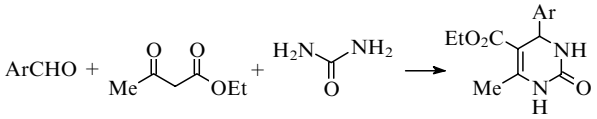
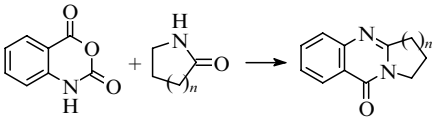
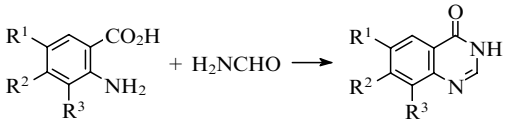
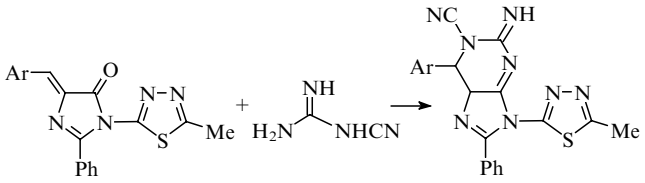
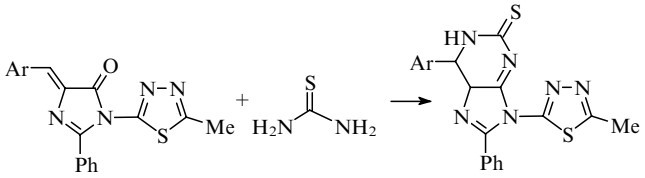
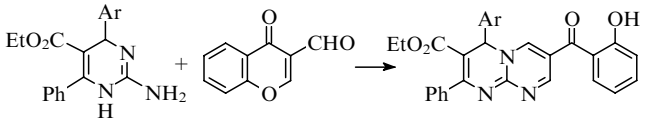
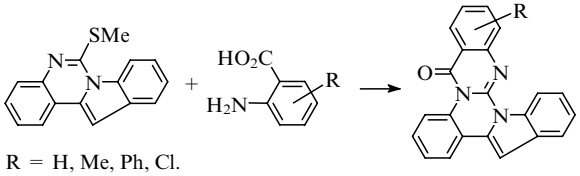
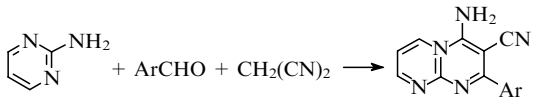
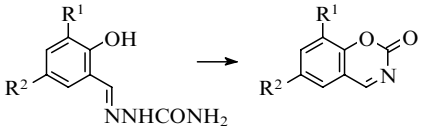
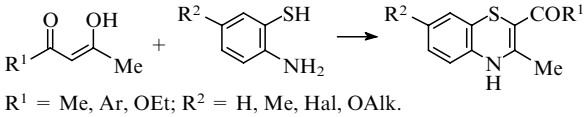
No.	Reaction	Conditions	Number of examples	Yield (%)	Ref.
3		SiO_2 or KSF,[a] or KH_2PO_4, H^+, MW, 1–6 min	17	8–94	308
4	 R = H, Alk.	SiO_2, MW, 5 min	5	>85	309
5	 R = H, Alk, Ar.	MW, 1.5–12 min	9	24–70	310
6	 R^1 = Me, CH_2Cl, Ph; R^2 = Me, Et.	MW, 2–6 min	7	77–93	311
7		$FeCl_3 \cdot 6H_2O$, MW, 4–6 min	8	87–95	312
8	 *n* = 1–3.	MW, 6–8 min	3	89–92	313
9	 R^1 = H, Alk, NO_2, Br, OH, OMe; R^2, R^3 = H, OMe, Br.	*a*) MW, 15–40 min *b*) Δ, 6 h	10 1	70–90 59	314 314
10		*a*) Al_2O_3, MW, 7–8 min *b*) Δ, 5–5.5 h	3 3	89–95 65–71	245 245
11		*a*) Al_2O_3, MW, 8–9 min *b*) Δ, 3.5–4 h	3 3	90–98 69–74	245 245
12		*a*) MW, 20 min *b*) EtOH, Δ, 4 h	5 5	>95 60–80	315 315
13	 R = H, Me, Ph, Cl.	graphite, MW, 30 min	4	14–90	316

Table 26 (continued).

No.	Reaction	Conditions	Number of examples	Yield (%)	Ref.
14		piperidine, MW, 3 min	1	85	317
15	 R^1, R^2 = H, Hal, OMe, NO_2.	K-10, MW, 1.5–3 min	10	83–94	318
16	 R^1 = Me, Ar, OEt; R^2 = H, Me, Hal, OAlk.	Al_2O_3, MW, 6–11 min	14	69–85	319

[a] Hereinafter, KSF is a support material.

ones were synthesised by the intramolecular cyclisation of substituted semicarbazones of salicylic aldehyde (Table 26, No. 15); the condensation of 2-aminothiophenols with β-diketones afforded 1,4-benzothiazines (No. 16).

The results compiled in Table 26 clearly demonstrate the superiority of the microwave activation over the thermal one.

d. Synthesis of six-membered heterocycles with three nitrogen atoms

Examples of the use of MWI in the synthesis of 1,2,4- and 1,3,5-triazines are given in Table 27.

One of the listed studies is worth a special note. Kidwai *et al.*[320] explicitly demonstrated the advantages of microwave activation as compared to thermal for 12 model reactions. Using alumina as a support they achieved quantitative yields of 1,2,4-triazines with simultaneous acceleration of the reactions by 3 orders of magnitude. This is a striking manifestation of the MWE action.

5. Heteroyclic compounds with 7 or more atoms in a ring

Results of the application of MWI to the synthesis of heterocyclic compounds containing $\geqslant 7$ atoms are summarised in Table 28. In particular, seven-membered heterocycles with two nitrogen atoms were prepared by the condensation of *o*-phenylenediamines with ketones in different catalytic environments (No. 1) and reaction of unsaturated amines with *N*-carboxyanthranilic anhydride (No. 2). The condensation of hydrazides with orthoesters yielded 4,7-dihydro-1,2,4-triazepin-8-one derivatives (No. 3). The reaction of *o*-aminothiophenols with α,β-unsaturated ketones gave rise to 2,3-dihydro-1,5-benzothiazepines (No. 4). A 8-membered dibenzodiazocinoquinazolinone was synthesised from 3-hydroxyquinazolinones and *N*-carboxyanthranilic anhydride (No. 5). The cyclocondensation of benzaldehydes with pyrrole afforded tetraarylporphyrins (No. 6). The cyclocondensation of phthalic acid or its derivatives was employed for the synthesis of phthalocyanines (No. 7). Cationic cyclophanes were afforded by the cyclocondensation of precyclophanes with *m*-terphenyl dibromides (No. 8). Two types of binuclear phthalocyanines (Nos 9 and 10) were

Table 27. Synthesis of 1,2,4- and 1,3,5-triazines.

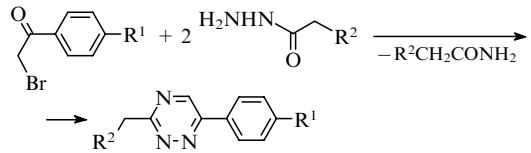
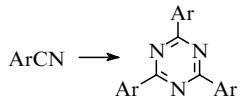
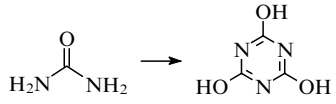
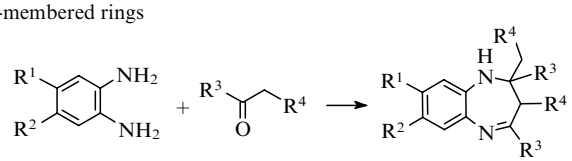
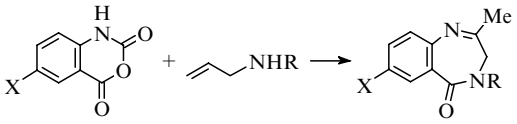
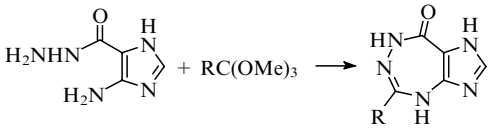
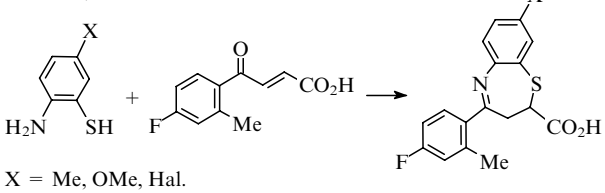
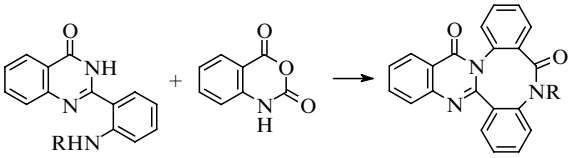
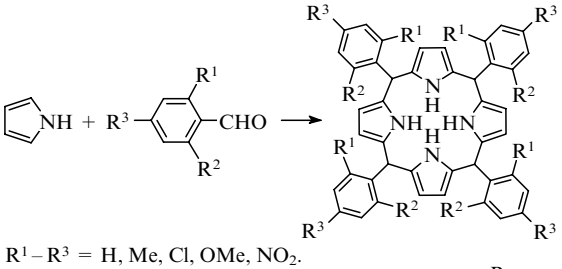
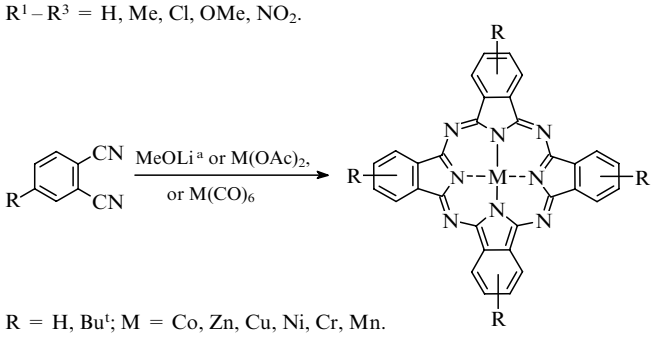
No.	Reaction	Conditions	Number of examples	Yield (%)	Ref.
1	 R^1 = H, Cl, NH_2; R^2 = Ar, OAr.	*a*) MW, 300–420 s	12	60–64	320
		b) SiO_2, MW, 90–120 s	12	70–79	320
		c) K-10, MW, 95–120 s	12	70–74	320
		d) Al_2O_3, MW, 60–95 s	12	88–95	320
		e) Δ, 18–25 h	12	50–59	320
2		*a*) $Yb(OTf)_3$ or Si(M) (M = Al, Zn, Ti), MW, 30–60 min	10	0–95	321
		b) Δ, 1–24 h	28	0–84	321
3		*a*) graphite, MW, 1–3 min	2	10–61	322
		b) Δ, 1–3 min	2	5–15	322

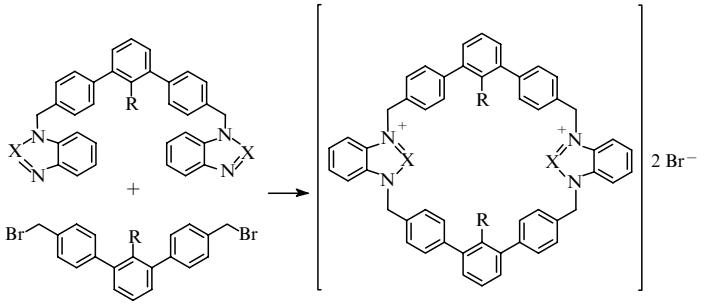
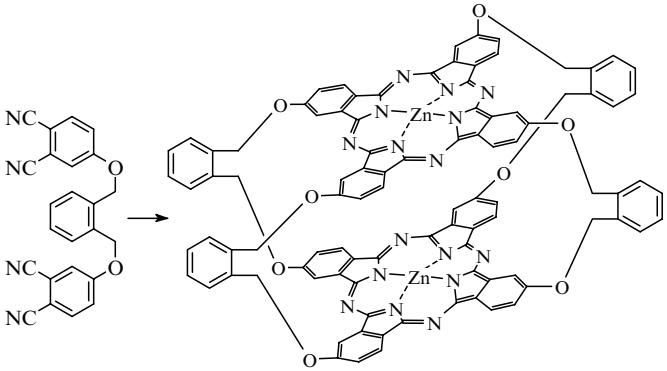
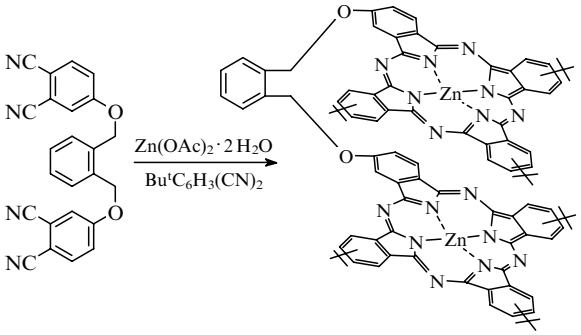
Table 28. Synthesis of heterocyclic compounds with 7 or more atoms in a ring.

No.	Reaction	Conditions	Number of examples	Yield (%)	Ref.
Seven-membered rings					
1	 R^1, R^2 = H, Alk, Ar, Cl, PhCO; R^3, R^4 = H, Alk, Ar.	*a*) Al_2O_3/P_2O_5, MW, 1 min *b*) AcOH, MW, 2–7 min	6 12	73–85 90–99	323 324
2	 R = Me, All; X = H, Hal.	*a*) MW, 5 min *b*) DMF, 60 min	6 6	55–69 27–45	325 325
3	 R = Alk, Ph.	*a*) EtOH, MW, 3 h *b*) Δ, 22 h	2 3	38–47 8–74	326 326
4	 X = Me, OMe, Hal.	*a*) KSF, MW, 9–15 min *b*) EtOH, HCl, Δ, 420–840 min	6 6	59–71 50–62	327 327
Eight-membered rings					
5	 R = H, Me, Et.	montmorillonite, MW, 7 min	2	72–73	328
16-Membered rings					
6	 R^1–R^3 = H, Me, Cl, OMe, NO_2.	$EtCO_2H$, MW, 3–5 min	8	4–43	329
7	 R = H, Bu^t; M = Co, Zn, Cu, Ni, Cr, Mn.	MW, 3–10 min	>15	<30–80 [b]	330

[a] $MeOLi$ or $M(OAc)_2$, or $M(CO)_6$

[b]

Table 26 (continued).

No.	Reaction	Conditions	Number of examples	Yield (%)	Ref.
8	 R = H, Br, CO_2H, CO_2Me; X = CR' (R′ = H, Me), N.	*a*) Na_2SO_4, MW, 13–15 min	10	30–60	331
		b) Δ, 5 days	10	40–70	331
9		*a*) $Zn(OAc)_2 \cdot 2H_2O$ (2 equiv.), *o*-$Cl_2C_6H_4$, MW, 15 min	1	6.8	332
		b) $Zn(OAc)_2 \cdot 2H_2O$ (20 equiv.), *o*-$Cl_2C_6H_4$, MW, 3 min	1	33	332
		c) iso-$C_5H_{11}OH$, Δ, 14 h	1	1.8	332
10		MW, 5 min	1	22[c]	333

[a] If lithium methoxide is used, a complex with two lithium atoms is formed; [b] if phthalic imide or anhydride or $RC_6H_3(CO_2H)_2$ in the presence of urea is used instead of $RC_6H_3(CN)_2$, the yields of products decrease; [c] the monosubstitution product is formed in a yield of 28%.

synthesised using the cyclocondensation of 1,2-bis(3,4-dicyanophenoxymethyl)benzene.

XV. Organophosphorus compounds

Alkylphosphonates were prepared by the microwave-assisted reaction of trialkyl phosphites with alkyl halides (the Arbuzov rearrangement) (Table 29, Nos 1 and 2) or aldehydes (the modified Arbuzov reaction) (No. 3). The Kabachnik–Fields reaction was utilised to synthesise aminoalkylphosphonates either under solvent-free conditions on a solid support (Nos 4 and 5) or using a catalyst in an ionic liquid (No. 6) or 1,2-dichloroethane (No. 7). The interaction of triphenylphosphine with alkyl halides gave rise to phosphonium salts (No. 8). The MWE is manifested in an increase in the yields of products in the cases of microwave activation (Nos 4 and 5).

XVI. Miscellaneous reactions

Table 30 lists several additional examples of microwave-assisted reactions not covered by Tables. 1–29.

XVII. Conclusion

The history of the utilisation of microwave technology in organic synthesis extends only over two decades. We may consider that it is now mainly in the stage of gathering empirical results. Despite the fact that the number of papers reporting the use of MWI for heating and activation of chemical reactions exceeded 2000, there is still no solid theoretical background underlying the experimentally observed specific microwave effect.

The above analysis of the published data indicates that the microwave method for the activation of chemical processes is applicable to nearly all known types of organic reactions. The net result of a microwave chemical reaction depends primarily on the nature of reactants (*i.e.*, on their structural features and distribution of the electron density therein), the reaction mechanism (the

Table 29. Synthesis of phosphonates, aminophosphonates and phosphonium salts.

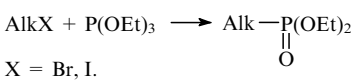
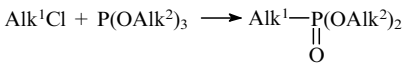
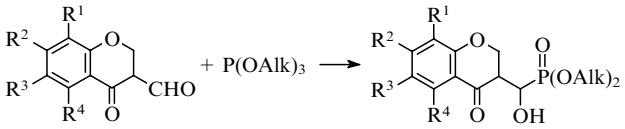
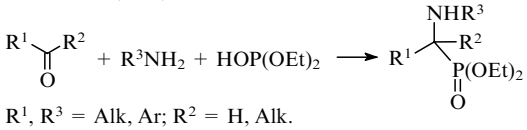
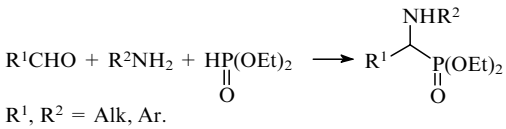
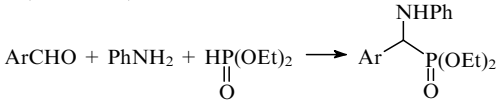
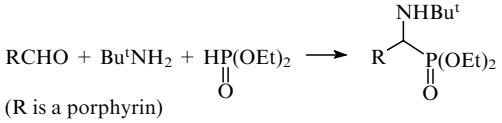
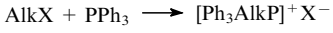
No.	Reaction	Conditions	Number of examples	Yield (%)	Ref.
1	 X = Br, I.	MW, 15–30 s	3	95–97	334
2		Al_2O_3, MW, 5–15 min	10	0–90	335
3	 R^1–R^4 = H, Alk, Cl.	Me_3SiCl, MW, 5–10 min	13	70–95	336
4	 R^1, R^3 = Alk, Ar; R^2 = H, Alk.	*a*) KSF, MW, 3–8 min *b*) PhMe, Δ, 5–12 h	18 18	75–91 65–80	337 337
5	 R^1, R^2 = Alk, Ar.	*a*) Al_2O_3, MW, 3–6 min *b*) Δ, 4–8 h	13 14	65–95 59–80	338 338
6		$Yb(OTf)_3$, $(bmim)BF_4$, MW, 2 min	6	83–100	339
7	 (R is a porphyrin)	CdI_2, $C_2H_4Cl_2$, MW, 9–10 min	4	80–85	340
8	 X = Br, I.	xylene, MW, 30 s	3	>99	334

Table 30. Miscellaneous reactions.

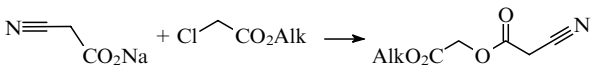
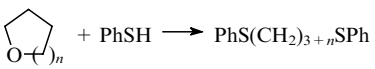
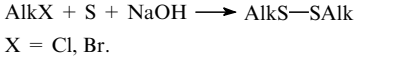
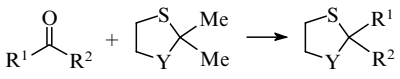
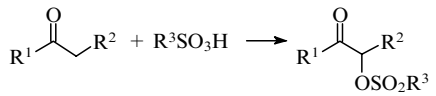
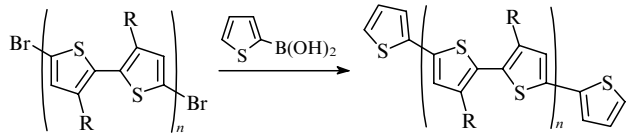
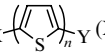
No.	Reaction	Conditions	Number of examples	Yield (%)	Ref.
1		Bu^n_4NBr/SiO_2, MW, 2–4 min	8	84–95	341
2	 n = 1, 2.	$InCl_3/SiO_2$, MW, 10–15 min	18	53–89	342
3	 X = Cl, Br.	PEG-400, DMF *a*) MW, 6–7 min *b*) Δ, 4 h	 9 9	 65–91 50–97	343
4	 R^1 = Alk, Ph; R^2 = H, Alk; Y = O, S.	KSF or Amberlyst 15, MW, 15 min	16	0–98	344

Table 30 (continued).

No.	Reaction	Conditions	Number of examples	Yield (%)	Ref.
5	 R^1 = Alk, Ar; R^2 = Alk; R^3 = OTs, $OC_6H_4NO_2$-*p*.	$PhI(OAc)_2$, MW, 10–40 s	32	75–95	345
6	 R = H, Me; *n* = 1, 2.	*a*) $Pd(OAc)_2$, Bu^n_4NBr, K_2CO_3, H_2O–PhMe, MW, 5 min	2	65–70	346
		b) [Pd],[a] KF/Al_2O_3, KOH, MW, 4–10 min	8 [b]	73–81	347

[a] Various Pd catalysts were used; [b] the synthesis of other polythiophenes with a common formula  (X, Y = H, Br; R = H, Me; *n* = 2–6) was also reported.

stronger the change in the polarity of the medium during the reaction, the more evident is the microwave effect), the duration and power of MWI, the types of supports, catalysts and solvents utilised. Therefore, the factors listed above have to be considered as a whole for any specific reaction type.

Numerous attempts have been undertaken (see, for instance, Refs 5–7,9,12,42) to reconcile the current theoretical concepts regarding the physics of microwave processes with the experimental results on organic syntheses performed under microwave activation. However, by no means all the factors affecting the overall output of a specific chemical reaction have been properly taken into account. In particular, the effects of MWI wavelength and power and support type on the direction, chemical and stereochemical yields of a chemical reaction are unknown up to date. Several studies emphasising these issues were published after 2002. In particular, Nüchter *et al.* [348] considered the best practices of microwave experiments, Kappe [349] and de la Hoz *et al.* [350] gave examples of successful applications of controlled MWI-heating to organic synthesis and Romanova *et al.* [351] described the influence of MWI on the direction and stereochemistry of reactions.

The rapidly increasing number of publications devoted to microwave syntheses clearly confirms the growing perception of MWI as a powerful tool in hands of organic chemists. The first monographs on MWI synthesis have already appeared.[352–354] Without doubt, the near future will witness expanding application of the method both on the laboratory and industrial scales, especially provided that its theoretical substantiation is elaborated. The efforts put into the optimisation of conditions of microwave organic syntheses will be definitely rewarded by the quickness and simplicity of experimental implementations, energy saving and cost efficiency intrinsic to MWI.

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