Review

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The synthesis of phosphinates: traditional *versus* green chemical approaches

Abstract: Three alternatives are discussed in comparison with the classical esterification of phosphinic chlorides by reaction with alcohols. All novel methods, such as microwave (MW)-assisted direct esterification, MW-assisted phase transfer catalyzed alkylating esterification and the propylphosphonic anhydride-promoted esterification, start from phosphinic acids and offer different advantages and disadvantages. The methods are analyzed from green chemical point of view.

Keywords: green chemistry; MW chemistry; phase transfer catalysis; phosphinates; phosphinic acids; T3P reagent.

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1 Introduction

The microwave (MW) technique has become an important tool in organic syntheses [1–3]. The advantages include shorter reaction times and higher yields, making more efficient preparations possible. MW irradiation may also promote reluctant reactions; moreover, otherwise impossible transformations may also take place under MW. In many cases, the reactions can be realized under solvent-free conditions. Acylations/esterifications, alkylations/substitutions/couplings, additions, eliminations/dehydrations and 2- or 3-component condensations are typical reactions that may be performed under MW conditions. The point of MW heating is that the local overheating effect appearing statistically in the bulk of the mixture makes efficient transformations possible. This may be understood if one considers the Arrhenius equation and the extent of local overheating that may be in the range of up to 60°C [3]. MW is especially suitable to promote reactions with a high enthalpy of activation. From industrial point of view, continuously operating MW reactors may be relevant [4, 5]. The reactors comprise parallel (thin) tubes made of glass. The reaction mixture must not be too viscous and heterogeneous. The utilization of the MW technique seems to be attractive also in organophosphorus chemistry [6–10].

In this paper, our recent results on the MW-assisted esterification of phosphinic acids are summarized and compared with other methods.

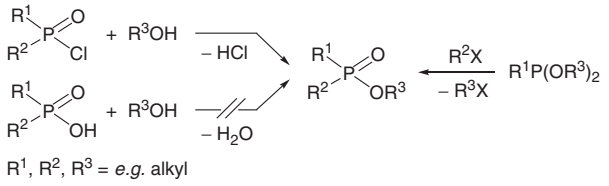
2 The synthesis of phosphinates by the reaction of phosphinic chlorides with alcohols

In most cases, phosphinates are prepared from phosphinic chlorides by reaction with alcohols (Scheme 1) [11, 12]. However, phosphinic chlorides are not inexpensive, the reaction is not atomic efficient and the hydrochloric acid formed is an environmental burden. Phosphinates may also be synthesized by the Arbuzov reaction [11]. It is well-known that phosphinic acids do not undergo direct esterification (Scheme 1).

Our target molecules were the alkoxy-phospholene oxides, alkoxy-phospholane oxides and alkoxy-hexahydrophosphinine oxides. The literature examples for the esterification of 1-halogeno-phospholene 1-oxides, mostly 1-chloro derivatives, are summarized in Table 1. Using the alcohols in the presence of triethylamine, or applying sodium methylate, both at room temperature or above, the yields were variable, and fall in the range of 27%–85% [13–17].

3 MW-assisted direct esterification and other derivatizations of cyclic phosphinic acids

It was a challenge for us to try to perform the direct esterification of phosphinic acids under MW conditions. The

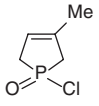
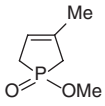
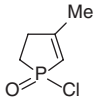
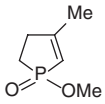
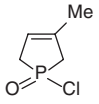
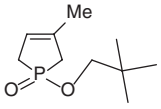
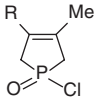
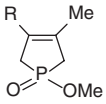
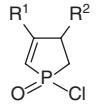
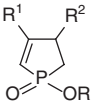
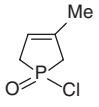
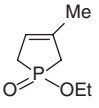
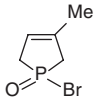
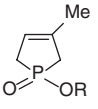


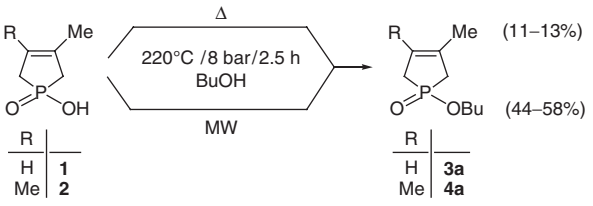
Scheme 1 Different approaches to phosphinates.

1-hydroxy-3-phospholene oxides (**1** and **2**) chosen as the starting phosphinic acids were, in the first approach, reacted with butanol as the esterifying component. Surprisingly, the cyclic phosphinic acids (**1** and **2**) underwent esterification above 200°C in a sealed vessel to give phosphinates **3** and **4** in yields of 44% and 58%, respectively. The comparative thermal experiments led only to low (~12%) conversions (Scheme 2) [18].

The esterifications were carried out using 15 equivalents of the alcohol in closed vessels equipped with a pressure controller tolerating a maximum pressure of 20 bar. For this, higher carbon atom chains, and hence less volatile alcohols were found to be the starting materials of choice. In these cases, we could work at reaction temperatures of 220°C–235°C and at overpressures of 1 bar–1.5 bar. Accordingly, the direct esterifications of cyclic phosphinic

Table 1 Literature examples for the esterification of halogeno-phospholene oxides.

Phosphinic chloride	Reagents solvent	Product	Yield (%)	References
	MeOH NEt_3 Et_2O		64	[13]
	MeOH NEt_3 Et_2O		62	[13]
	NeopentylOH NEt_3 PhH		85	[14]
	NaOMe PhMe		R | H | 80 Me | 81	[15]
	ROH NEt_3 CH_2Cl_2		R^1 | R^2 | R | H | H | Me | 27 H | H | Et | 52 Me | H | Et | 72 Me | H | Bu | 36 Me | H | Hex | 48 Me | H | Dodec | 43 Me | Me | Me | 45 Me | Me | Et | 47 Me | Me | Hex | 45	[16]
	EtOH NEt_3 CH_2Cl_2		47	[16]
	ROH NEt_3 Et_2O		R | Me | 32 Et | 45 Pr | 29 Bu | 61 Pent | 70 ^{i}Hex | 46 Oct | 53	[17]



Scheme 2 Direct esterification of 1-hydroxy-3-phospholene oxides with BuOH.

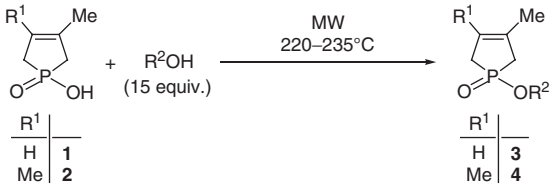
acids **1** and **2** were also carried out with *n*-pentanol, isopentanol, *n*-octanol, 2-ethylhexanol (isooctanol) and dodecyl alcohol, to afford phosphinates **3** and **4**, with one exception, in yields of 67%–95% (Scheme 3) [19–21].

The direct esterification was then extended to 1-hydroxy-phospholane oxides. Esterification of the 3-methyl derivative (**5**) at 220°C–235°C provided the corresponding phosphinates (**6**) as a 1:1 mixture of two diastereomers in yields of 59%–86% (Scheme 4) [21].

The analogous 3,4-dimethyl derivatives (**8**) were obtained from the two isomers of the starting phosphinic acid (**7**) as a mixture of three isomers (**8A**, $\mathbf{8B_1}$ and $\mathbf{8B_2}$). The yields fall in the range of 50%–72% (Scheme 5) [21].

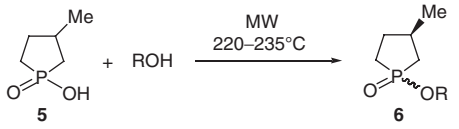
Finally, 1-hydroxy-3-methyl-1,2,3,4,5,6-hexahydrophosphinine 1-oxide **9** was subjected to direct esterification with octanols to provide the cyclic phosphinates **10** as a mixture of two diastereomers (Scheme 6) [21].

Then, the energetic background for the esterification of phosphinic acids was studied by B3LYP/6-31++G(d,p) calculations. As a comparison, the esterification of acetic acid with methanol was also calculated. All esterifications were found to be thermoneutral, implying a slight exothermicity or endothermicity. However, the requirement for the enthalpy of activation was much higher for the esterification



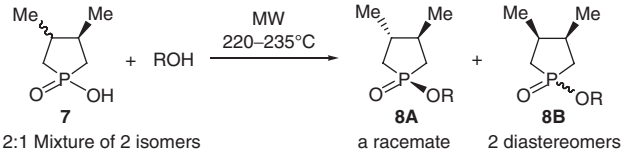
R^1	R^2	T (°C)	t (h)	Conversion (%)	Yield of the esters (%)
H	$^nC_5H_{11}$ (**b**)	220	2.5	100	94 (**3b**)
H	$^iC_5H_{11}$ (**c**)	235	3	100	74 (**3c**)
H	$^nC_8H_{17}$ (**d**)	220	2	100	71 (**3d**)
H	$^iC_8H_{17}$ (**e**)	220	2	100	76 (**3e**)
Me	$^nC_5H_{11}$ (**b**)	235	3	90	67 (**4b**)
Me	$^iC_5H_{11}$ (**c**)	235	4	80	57 (**4c**)
Me	$^iC_8H_{17}$ (**e**)	220	2.5	100	82 (**4e**)
Me	$C_{12}H_{25}$ (**f**)	230	2	100	95 (**4f**)

Scheme 3 MW-assisted direct esterification of 1-hydroxy-3-phospholene oxides with different alcohols.



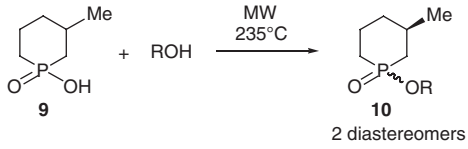
R	T (°C)	t (h)	Isomeric composition (%)	Yield of the esters (%)
$^nC_5H_{11}$ (**b**)	235	3	1:1	79 (**6b**)
$^iC_5H_{11}$ (**c**)	235	4	1:1	59 (**6c**)
$^nC_8H_{17}$ (**d**)	230	4	1:1	72 (**6d**)
$^iC_8H_{17}$ (**e**)	220	3	1:1	86 (**6e**)

Scheme 4 MW-assisted direct esterification of 1-hydroxy-3-methylphospholane oxide.



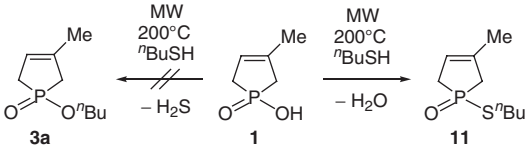
R	T (°C)	t (h)	Isomeric composition (%)	Yield of the esters (%)
$^nC_5H_{11}$ (**b**)	235	6	70 - 15 - 15	60 (**8b**)
$^iC_5H_{11}$ (**c**)	235	5	64 - 19 - 17	56 (**8c**)
$^nC_8H_{17}$ (**d**)	230	4	~70 - 15 - 15	72 (**8d**)
$^iC_8H_{17}$ (**e**)	220	4	66 - 19 - 15	50 (**8e**)

Scheme 5 MW-assisted direct esterification of 1-hydroxy-3,4-dimethylphospholane oxide.



R	t (h)	Isomeric composition (%)	Yield of the esters (%)
$^nC_8H_{17}$ (**d**)	4	66 - 34	62 (**10d**)
$^iC_8H_{17}$ (**e**)	6	69 - 31	54 (**10e**)

Scheme 6 MW-assisted direct esterification of 1-hydroxy-3-methyl-1,2,3,4,5,6-hexahydrophosphinine oxide.



Scheme 7 MW-assisted direct esterification with thiobutanol.

of phosphinic acids (102 kJ mol^{-1}–140 kJ mol^{-1}) than that for the esterification of acetic acid (75 kJ mol^{-1}) [20].

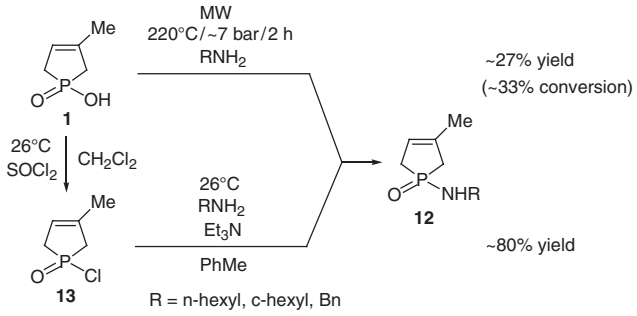
The esterification of 1-hydroxy-3-methyl-3-phospholene oxide (**1**) was also studied with thioalcohols. Using thiobutanol, the thiobutoxy-phospholene oxide (**11**) could be obtained in a yield of 38%. However, the butoxy-product (**3a**) was not formed (Scheme 7) [22].

The outcome of the reaction proved that in the above type direct esterifications indeed the alcohol is phosphinoylated and not the phosphinic acid is alkylated. The conversion and yield were, however, moderate; that was justified by quantum chemical calculations. It was found for the above model that the transformation is endothermic (48.5 kJ mol^{-1}), and the enthalpy of activation is higher (145 kJ mol^{-1}) than that for the reaction with butanol (102 kJ mol^{-1}) [22]. This explains why the conversion was incomplete even under MW conditions.

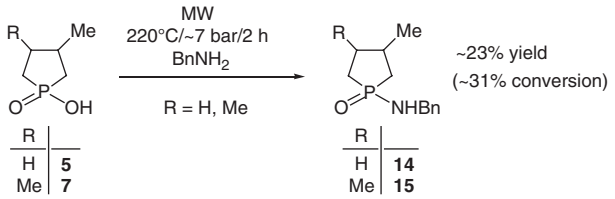
On the basis of the above experiences, it was of interest to try also MW-assisted direct amidations of 1-hydroxy-3-methyl-3-phospholene oxide (**1**). Using hexylamines and benzylamine at 220°C, the amidations took place in low (approximately 33%) conversions. It was better to synthesize the cyclic phosphinic amides (**12**) by the classical reaction sequence involving the corresponding phosphinic chloride (**13**) as the intermediate. In this way, the amides (**12**) were obtained in approximately 80% yields (Scheme 8) [23].

In the MW-assisted direct amidation of 1-hydroxyphospholane oxides **5** and **7**, similarly low conversions were experienced as for the amidation of 1-hydroxy-3-phospholene oxide **1** (Scheme 9) [23].

Quantum chemical calculations suggested that the direct amidations are also endothermic, but the values for the enthalpy of activation are not so high [23].



Scheme 8 Methods for the amidation of 1-hydroxy-3-methyl-3-phospholene oxide.



Scheme 9 MW-assisted direct amidation of 1-hydroxyphospholane oxides.

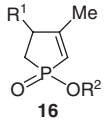
The following may be concluded on MW *versus* thermal heating and on the scope and limitation of MW-assisted reactions:

1. The role of MW is to enhance reactions with higher (≥100 kJ mol^{-1}) enthalpy of activation.
2. This is possible due to the statistically occurring local overheating effect (that may be up to 60°C) in the bulk of the reaction mixture.
3. Hence, there is no "black magic" around the effect of MWs. If the reaction discussed takes place only to the effect of MW irradiation (and not with conventional thermal heating), one can speak about a special MW effect.
4. The MW assistance is optimal for thermoneutral reactions. Endothermicity works against the beneficial effect of MW.

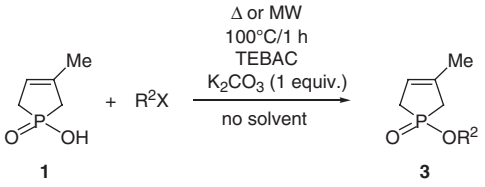
4 MW-assisted alkylating esterification of cyclic phosphinic acids

Another possibility for the synthesis of phosphinic esters involves the alkylation of phosphinic acids. This approach was utilized in the esterification of 1-hydroxy-3-methyl-3-phospholene oxide (**1**) using alkyl halides as the alkylating agents in the presence of 1 equivalent K_2CO_3 as the base and 5% triethylbenzylammonium chloride (TEBAC) as the phase transfer catalyst under solvent-free conditions. At a reaction temperature of 100°C, the butyl and benzyl esters of the methyl-hydroxy-phospholene oxide (**3a** and **3g**) were obtained in yields of 96% and 94%, respectively. It is obvious from the data of Scheme 10 that both MW and the presence of the catalyst are beneficial in the case of butyl bromide as the alkylating agent. At the same time, using benzyl bromide, there is no need to apply TEBAC [19, 24]. This can be explained by the fact that, in the case of alkyl halides with normal reactivity, the use of phase transfer catalysis and MW irradiation is beneficial, as the effects are synergistic, while in the case of alkyl halides with increased reactivity, the use of MW is enough in itself.

Isomerized by-product **16** could also be detected in the reaction mixture in a small quantity.



16
≤ 2%



1 3

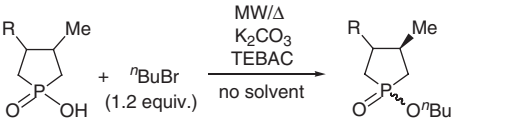
R^2X	TEBAC	Mode of heating	Yield of the esters (%)
^{n}BuBr (**a**)	–	Δ	64 (**3a**)
^{n}BuBr (**a**)	5%	Δ	89 (**3a**)
^{n}BuBr (**a**)	–	MW	69 (**3a**)
^{n}BuBr (**a**)	5%	MW	96 (**3a**)
BnBr (**g**)	–	Δ	85 (**3g**)
BnBr (**g**)	5%	Δ	87 (**3g**)
BnBr (**g**)	–	MW	92 (**3g**)
BnBr (**g**)	5%	MW	94 (**3g**)

Scheme 10 Alkylating esterification of 1-hydroxy-3-methyl-3-phospholene oxide.

Then, the alkylating esterification was extended to the preparation of 1-butoxy-phospholane oxides. The above mentioned observations are valid both for the esterification of the monomethyl- and dimethyl-1-hydroxyphospholane oxides (**5** and **7**, respectively). According to this, the combined application of the MW and the phase transfer catalytic technique was justified at 100°C, with butyl bromide as the alkylating agent (Scheme 11) [19, 24].

At the same time, at a higher reaction temperature of 120°C, the use of TEBAC became unnecessary.

Finally, 1-hydroxy-3-methyl-1,2,3,4,5,6-hexahydrophosphinine oxide (**9**) was subjected to alkylating esterification with butyl bromide. The experiences were similar to those for phospholane oxides (Scheme 12) [19, 24].

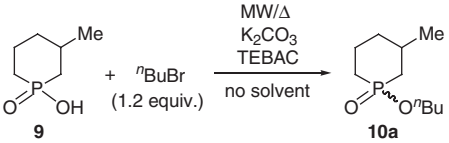


R	
H	**5**
Me	**7**

R		Composition
H	**6a**	1:1 mixture of two isomers
Me	**8a**	3:1:1 mixture of three isomers

R	TEBAC	Mode of heating	T (°C)	t (h)	Yield of the esters (%)
H	–	Δ	100	2	22 (**6a**)
H	5%	Δ	100	2	80 (**6a**)
H	–	MW	100	2	77 (**6a**)
H	5%	MW	100	1.5	95 (**6a**)
H	–	MW	120	2	95 (**6a**)
H	5%	MW	120	1	94 (**6a**)
Me	–	Δ	100	2	16 (**8a**)
Me	5%	Δ	100	2	75 (**8a**)
Me	–	MW	100	2	46 (**8a**)
Me	5%	MW	100	1.5	98 (**8a**)
Me	–	MW	120	2	93 (**8a**)
Me	5%	MW	120	1	94 (**8a**)

Scheme 11 Alkylating esterification of 1-hydroxyphospholane oxides.



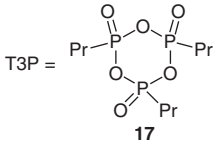
9 **10a**
1:1 mixture of two isomers

TEBAC	Mode of heating	T (°C)	t (h)	Yield of **10a** (%)
–	Δ	100	1	41
5%	Δ	100	1	66
–	MW	100	3	64
5%	MW	100	1	73
–	MW	120	1	70
5%	MW	120	1	81

Scheme 12 Alkylating esterification of 1-hydroxy-3-methyl-1,2,3,4,5,6-hexahydrophosphinine oxide.

5 Esterification of cyclic phosphinic acids in the presence of the T3P reagent

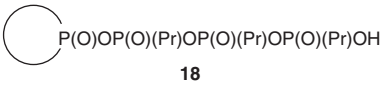
After the MW-assisted direct esterification and alkylating esterification of phosphinic acids, a third possibility was also studied. This is the propylphosphonic anhydride (T3P)-promoted esterification of a few cyclic phosphinic acids. T3P is a versatile reagent in a number of condensation reactions including different acylations [25].



17

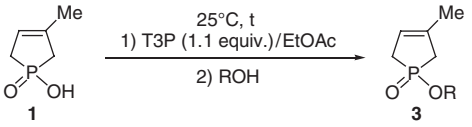
It was found that T3P was extremely useful in promoting the esterification between cyclic phosphinic acids **1**, **2** and **5** as well as **7** and different alcohols already at 25°C to furnish the corresponding phosphinates **3**, **4a**, **6a** and **8a**, as shown in Schemes 13–16, respectively [26].

The only problem is that the T3P reagent must be used in a quantity of 1.1 equivalents. Hence, it is not a catalyst, but a reactant. T3P reacts with the phosphinic acids to give the corresponding anhydride (in general **18**) that is already reactive enough to attack the alcohols.



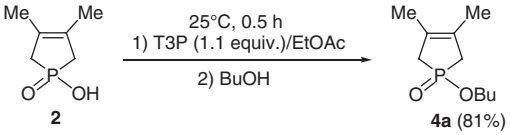
18

It can be seen that T3P makes efficient esterifications possible, however, its application means extra cost and the reaction is not atomic efficient.

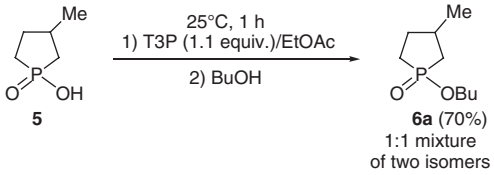


R		Time (h)	Yield of the esters (%)
Me	(**h**)	0.5	80 (**3h**)
Et	(**i**)	0.5	77 (**3i**)
Pr	(**j**)	0.5	80 (**3j**)
^{i}Pr	(**k**)	3	76 (**3k**)
Bu	(**a**)	0.5	82 (**3a**)
^{i}Bu	(**l**)	0.5	86 (**3l**)
^{s}Bu	(**m**)	3	88 (**3m**)
Pent	(**b**)	0.5	94 (**3b**)
^{i}Pent	(**c**)	0.5	91 (**3c**)
^{c}Hexyl	(**n**)	2	89 (**3n**)
Bn	(**g**)	0.5	95 (**3g**)

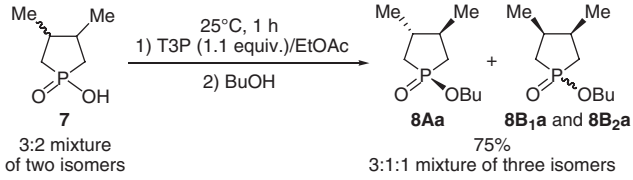
Scheme 13 Esterification of 1-hydroxy-3-methyl-3-phospholene oxide in the presence of the T3P reagent.



Scheme 14 Esterification of 1-hydroxy-3,4-dimethyl-3-phospholene oxide in the presence of the T3P reagent.



Scheme 15 Esterification of 1-hydroxy-3-methylphospholane oxide in the presence of the T3P reagent.



Scheme 16 Esterification of 1-hydroxy-3,4-dimethylphospholane oxide in the presence of the T3P reagent.

6 Conclusions

It is known that phosphinates are most often prepared by the reaction of phosphinic chlorides with alcohols in organic solvents at/or above room temperature. This reaction takes place with the formation of a hydrochloric acid by-product with two consequences: a base has to be used and the atomic efficiency is typically <80%. The MW-assisted direct esterification of phosphinic acids offers advantages, such as the use of halogen-free reagents, solvent-free accomplishment and a quite good atomic efficiency. However, problems include the relatively high reaction temperature of approximately 215°C required and the lack of suitable larger-scale MW reactors. The third method, the MW-promoted phase transfer catalytic alkylation of phosphinic acids under solvent-free conditions is basically attractive, but the not "green" reagents, alkyl halides are costly, K_2CO_3 has to be used as the base, and the atomic efficiency is only medium. Finally, the esterification of phosphinic acids with alcohols in the presence of T3P is an excellent method, as it requires mild conditions and gives the phosphinates in high yields. However, the expensive T3P reagent has to be used in a 1.1 equivalents quantity that results in a low atomic efficiency, due to the formation of the by-product. It is concluded that there is no method that can be regarded as the best, but all kinds of preparation have advantages and disadvantages.

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