IR Temperature Settings for High-Throughput Rotors

Introduction

The small-scale high-throughput rotors

- Rotor 4x48 MC Wellplate
- Rotor 4x20MGC
- Rotor 4x24MG5
- Rotor 64MG5

have been established as reliable tools for the individual stages of parallel synthesis. Reaction screening of as much as 192 experiments in one run as well as parallel optimizations and gram-scale library generation with up to 96 samples can be accomplished.

Multiwave PRO High-Throughput Rotors

All rotor types can be used up to 200 °C and 20 bar. They are straightforward to employ without immersing temperature probes or pressure sensors. Reaction control is achieved simply by IR temperature control and power regulation. By setting appropriate IR limits, the user has to take precautions not to exceed the temperature and pressure limits of these high-throughput rotors.

Whereas the temperature stability of the equipment would not be a major concern, the pressure limitation is the more crucial value, especially for the glass vials of Rotor 4x20MGC, Rotor 4x24MG5, and Rotor 64MG5, respectively. Various solvents with distinct boiling points and individual vapor pressure will reach the maximum pressure of 20 bar at different temperatures (see Table 1). Therefore, especially low-boiling solvents must not be used at too high temperatures as the 20 bar limit would be exceeded.

Since the surface temperatures of glass vials (Rotor 64MG5) or silicon carbide plates are measured the real reaction temperature inside the vials/wells is different and it will also be dissimilar in the individual rotor types. Thus an IR temperature of e.g. 150 °C will result in differing reaction temperatures inside the vials in the various rotors.

Therefore correlation factors (f_C) have been determined for each rotor, which allow calculating the current reaction temperature from the measured IR value. This correlation factor is an average value from several organic solvents monitored over the most relevant temperature range from 100–200 °C. Multiplying the measured IR value with f_C gives the reaction temperature in good approximation.

As a crude rule of thumb a reaction temperature 10% higher than the measured IR value can be assumed. This leads to satisfactory results for most reactions.

As a guideline we present in the following table the appropriate operational limits of several common organic solvents used in (microwave-assisted) synthesis for the individual rotor types. Within these limits the stability of the reaction containers will not be exceeded and the experiments are conducted safely.

Note: Solvents with a boiling point above 100 °C reach the 20 bar limit noticeably beyond 200 °C, thus it is acceptable to use these solvents (all entries following water in Table 1) at an IR limit of 180 °C in all rotors, although the inside temperature will reach up to approximately 220 °C (in Rotor 64MG5).

Table 1. Operational IR Temperature Limits of common solvents:

Solvent	Boiling Point	Temperature @ 20 bar	Pressure @ 200 °C	Rotor 4x48MC f_C = 1.103	Rotor 4x20MGC* f_C = 1.148	Rotor 4x24MG5 f_C = 1.152	Rotor 64MG5 f_C = 1.214
Dichloromethane (DCM)	40 °C	163 °C	36.1 bar	147 °C	141 °C	141 °C	134 °C
Acetone	56 °C	180 °C	28.1 bar	163 °C	156 °C	156 °C	148 °C
Chloroform	61 °C	193 °C	22.2 bar	175 °C	167 °C	167 °C	159 °C†
Methanol	65 °C	166 °C	40.5 bar	150 °C	144 °C	144 °C	136 °C
THF	65 °C	200 °C	20.0 bar	180 °C	173 °C	173 °C	165 °C
Hexane	69 °C	207 °C	18.0 bar	180 °C	173 °C	173 °C	165 °C†
Ethyl Acetate	77 °C	208 °C	17.7 bar	180 °C	173 °C	173 °C	165 °C
Tetrachloromethane	77 °C	222 °C	14.6 bar	180 °C	175 °C	175 °C	165 °C†
Ethanol	78 °C	181 °C	29.7 bar	163 °C	157 °C	157 °C	149 °C
Acetonitrile	81 °C	214 °C	15.9 bar	180 °C	175 °C	175 °C	165 °C
Cyclohexane	81 °C	226 °C	13.5 bar	180 °C	175 °C	175 °C	165 °C†
2-Propanol	82 °C	187 °C	25.9 bar	169 °C	162 °C	162 °C	154 °C
Dichloroethane (DCE)	84 °C	221 °C	14.3 bar	180 °C	175 °C	175 °C	165 °C
Dimethoxyethane (DME)	84 °C	218 °C	14.9 bar	180 °C	175 °C	175 °C	165 °C
Water	100 °C	213 °C	15.5 bar	180 °C	175 °C	175 °C	165 °C
Dioxane	101 °C	242 °C	10.2 bar	180 °C	175 °C	175 °C	170 °C†
Formic Acid	101 °C	230 °C	11.3 bar	180 °C	175 °C	175 °C	170 °C
Nitromethane	101 °C	236 °C	10.6 bar	180 °C	175 °C	175 °C	170 °C
Toluene	111 °C	263 °C	7.5 bar	180 °C	180 °C	180 °C	180 °C†
Acetic Acid	118 °C	249 °C	8.0 bar	180 °C	180 °C	180 °C	180 °C
1-Butanol	118 °C	231 °C	9.7 bar	180 °C	180 °C	180 °C	180 °C
Chlorobenzene	132 °C	292 °C	4.7 bar	180 °C	180 °C	180 °C	180 °C
Xylene	140 °C	299 °C	4.0 bar	180 °C	180 °C	180 °C	180 °C†
Dimethylformamide (DMF)	154 °C	325 °C	2.8 bar	180 °C	180 °C	180 °C	180 °C
Dimethylacetamide (DMA)	165 °C	325 °C	2.3 bar	180 °C	180 °C	180 °C	180 °C
1,2-Dichlorobenzene	179 °C	359 °C	1.6 bar	180 °C	180 °C	180 °C	180 °C
Dimethylsulfoxide (DMSO)	189 °C	310 °C	1.4 bar	180 °C	180 °C	180 °C	180 °C
Ethylene glycol	197 °C	337 °C	1.1 bar	180 °C	180 °C	180 °C	180 °C
N-Methylpyrrolidone (NMP)	204 °C	375 °C	1.0 bar	180 °C	180 °C	180 °C	180 °C
Nitrobenzene	210 °C	387 °C	1.0 bar	180 °C	180 °C	180 °C	180 °C

*temperatures only applicable if the aluminum assembly is used to set up the vials (see reference manual D49IB001 for details)

†maximum allowed temperature hardly reachable in Rotor 64MG5, since solvent is a poor microwave absorber