**Table 1 | Reaction optimisation**

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| --- | --- | --- |
| Entry | Change from above conditions | Yield (%) |
| **1** | **none** | **87(81)** |
| 2 | THF instead of 1,2-DME | 84(78) |
| 3 | Ni(NO3)2**.**6H2O as the catalyst | 69 |
| 4 | NiCl2**.**6H2O as the catalyst | 67 |
| 5 | NiCl2 with H2O (3.0 equiv) | 54 |
| 6 | no Ni catalyst | 0 |
| 7 | no Zn | 0 |
| 8 | no ZnCl2 | 0 |
| 9 | Mn instead of Zn | 49 |
| 10 | MgCl2 instead of ZnCl2 | 18 |
| 11 | 1.0 equiv of **2** | 41 |
| 12 | open atmosphere | 66 |
| 13 | 12 h | 68 |
| 14 | 10 mol% of Ni catalyst | 68 |
| 15 | 4-methoxybenzoyl chloride | 0 |
| 16 | 4-methoxybenzaldehyde | 0 |
| 17 | 2-mercaptopyridine, 20 mol% | 65 |
| 18 | 2,2′-dipyridyl disulfide, 20 mol% | (48) |



Reaction conditions: **1** (0.20 mmol), **2** (0.30 mmol). Yields were determined by GC using dodecane as the internal standard. Isolated yields are given in parentheses. 1,2-DME = 1,2-dimethoxyethane; PMP = *para*-methoxyphenyl.

Table 2 | Scope of the nickel-catalysed hydroacylation



Reaction conditions: *a*Thioester (0.20 mmol), alkyne (1.5 equiv), 1,2-DME. *b*Thioester (0.20 mmol), alkyne (1.5 equiv), THF, 2,2′-dipyridyl disulfide (20 mol%). Isolated yields.