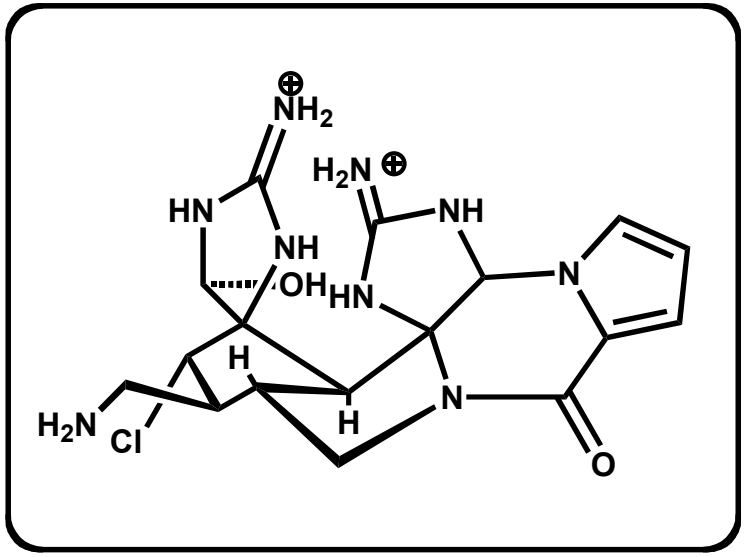
Total synthesis of Palau'amine



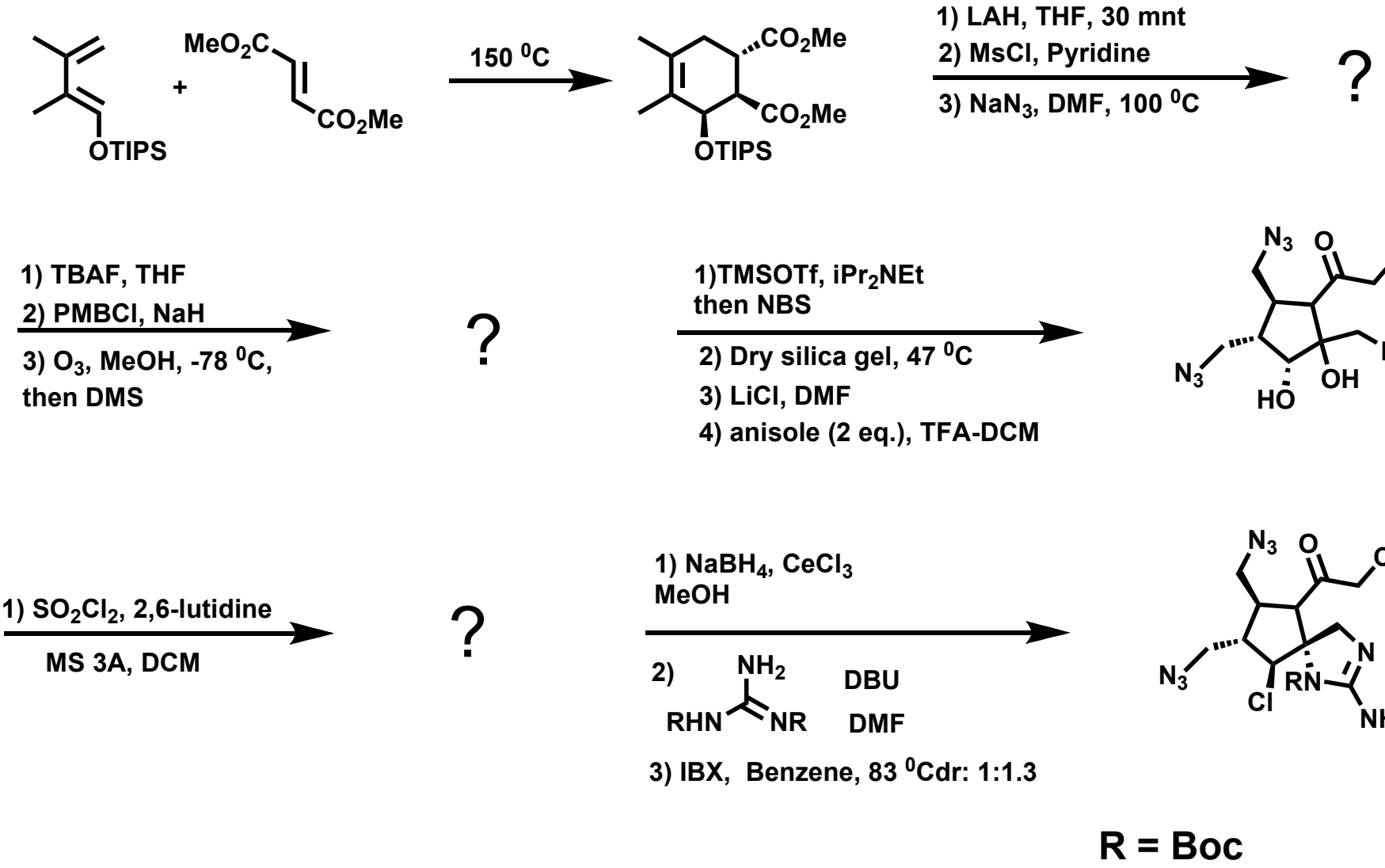
(±) Palau'amine

Phil S. Baran and co-workers

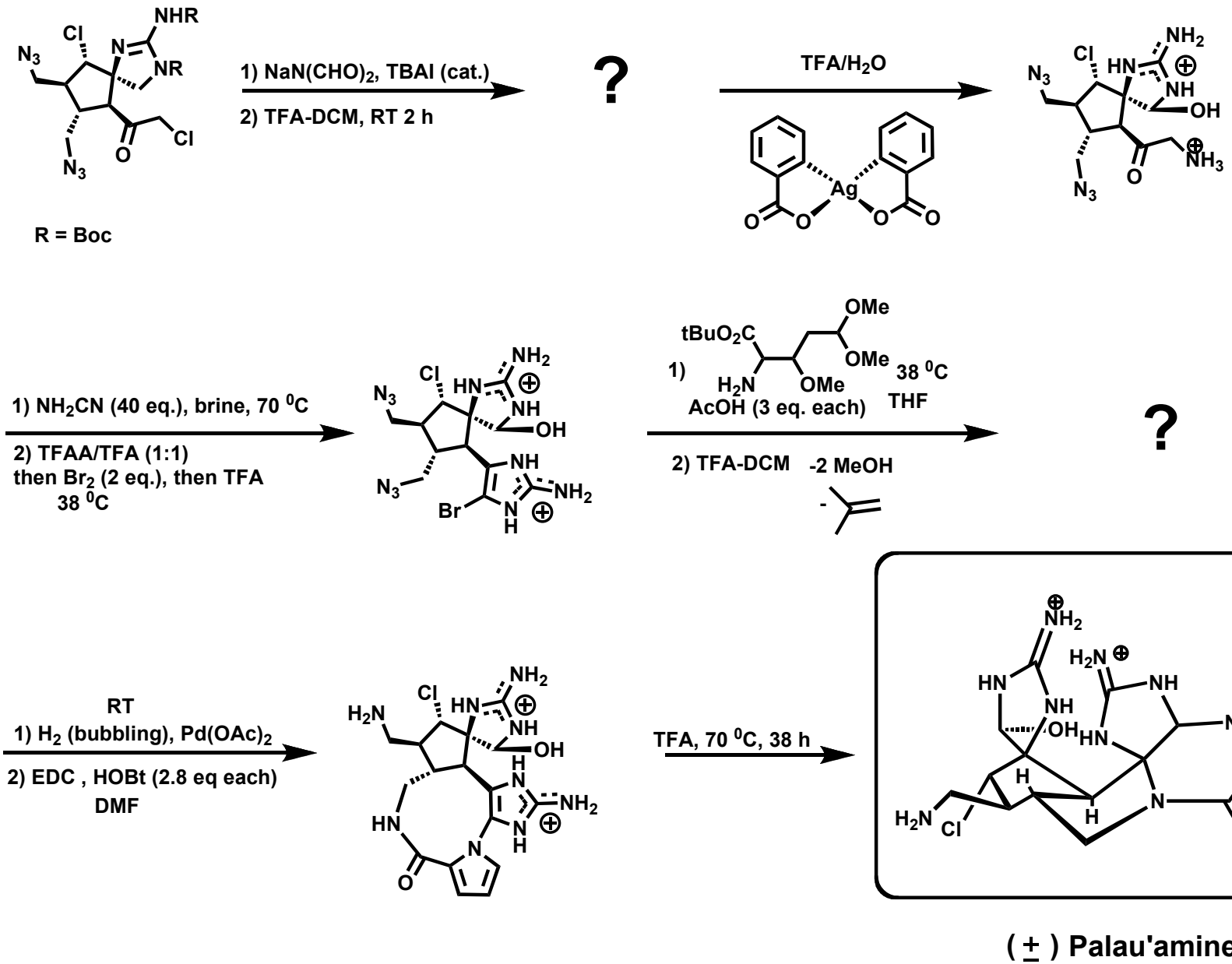
References..

Highlighted in ***Angew. Chem. Int. Ed. 2010, 49, 2972 – 2974***

Angew. Chem. Int. Ed. 2010, 49, 1095 –1098


MeO2C
CO2Me
OTIPS
150 0C
CO2Me
CO2Me
OTIPS
1) LAH, THF, 30 mnt
2) MsCl, Pyridine
3) NaN3, DMF, 100 0C
?
1) TBAF, THF
2) PMBCl, NaH
3) O3, MeOH, -78 0C,
then DMS
?
1)TMSOTf, iPr2NEt
then NBS
2) Dry silica gel, 47 0C
3) LiCl, DMF
4) anisole (2 eq.), TFA-DCM
N3
O
Cl
Br
N3
OH
HO
1) SO2Cl2, 2,6-lutidine
MS 3A, DCM
?
1) NaBH4, CeCl3
MeOH
2)
NH2
RHN
NR
DBU
DMF
3) IBX, Benzene, 83 0Cdr: 1:1.3
N3
O
Cl
N3
Cl
RN
N
NHR

R = Boc


NHR
Cl
N
NR
N3
N3
Cl
O
R = Boc
1) NaN(CHO)2, TBAI (cat.)
2) TFA-DCM, RT 2 h
?
TFA/H2O
Ag
O
O
O
O
NH2
Cl
HN
NH
OH
N3
O
NH3
N3
1) NH2CN (40 eq.), brine, 70 0C
2) TFAA/TFA (1:1)
then Br2 (2 eq.), then TFA
38 0C
NH2
Cl
HN
NH
OH
N3
NH
NH2
N3
Br
N
H
tBuO2C
OMe
OMe
1)
H2N
OMe
38 0C
AcOH (3 eq. each)
THF
2) TFA-DCM
-2 MeOH
-
?
RT
1) H2 (bubbling), Pd(OAc)2
2) EDC , HOBt (2.8 eq each)
DMF
NH2
Cl
HN
H2N
NH
OH
H
N
NH2
HN
N
H
N
O
TFA, 70 0C, 38 h
NH2
H2N
HN
NH
NH
OH
HN
NH
N
H
H2N
Cl
H
N
O

(±) Palau'amine