A novel multicomponent microwave-assisted synthesis of 5-aza-adenines
(Supplementary Information)
7-Amino-1,2,4-triazolo[1,5-a][1,3,5]triazine (5a)
13C DEPTQ-135 with IH Decoupling

Current Data Parameters
NAME AC-GE1- warp
EXP NO 2
PROC NO 1
F2 - Acquisition Parameters
Data 212011
Time 8.29
INSTRM spect
PROBD 5 mm PABBO BB-
EPIFLGD deepgaga
TD 65.56
EOUENT DROZO
NS 164
DS 4
SNR 220.08, 824 Hz
FRES 0.33092 Hz
AQ 1.4855326 sec
AG 203
DW 22.667 usec
DE 9.000 usec
TE 299.8 K
FSL1 11.5000000
FSL1 0.00000000 sec
F1 1.0000000 sec
F2 0.0000000 sec
FSL1 0.0000000 sec
FSL1 1.0000000 sec

-------- CHANNEL F1 --------
NUC1 13C
F1 10.00 usec
F13 2000.00 usec
FLM1 54.6940024 W
FSL1 100.002358 Hz
SPM1 0.49000004 Hz
SPM2 0.50000000 Hz
SPM3 8,662,1997 W

-------- CHANNEL F2 --------
UPPER2 100.00000000 W
UPPER2 0.49000004 Hz

-------- GRADIENT CHANNEL --------
UPRM1 0.8928, 0.31
UPRM2 0.8928, 0.32
UPRM3 0.8928, 0.32
UPRM4 31.00 Hz
UPM2 31.00 Hz
UPM3 31.00 Hz
UPM4 1000.00 usec

F2 - Processing parameters
SI 65.56
DF 100.002358 Hz
MDW EM
BBB 0
LB 1.00 Hz
UB 0
HC 1.40
7-Amino-2-phenyl-1,2,4-triazolo[1,5-a][1,3,5]triazine (5b)
13C DEPTQ-135 with IH Decoupling
7-Amino-2-(3-fluorophenyl)-1,2,4-triazolo[1,5-a][1,3,5]triazine (5c)

7.41 (dt, J=2.69, 8.58 Hz, 1 H)
7.64 (td, J=8.00, 6.00 Hz, 1 H)
7.92 (ddd, J=1.39, 2.55, 9.97 Hz, 1 H)
8.06 (td, J=1.12, 7.71 Hz, 1 H)
Avance III – 13C DEPTQ-135 with IH Decoupling

113.41 (d, J=23.30 Hz)
117.53 (d, J=21.06 Hz)
123.05 (d, J=2.71 Hz)
131.18 (d, J=8.28 Hz)
132.48 (d, J=8.41 Hz)
162.28 (d, J=3.06 Hz)
162.32 (d, J=243.90 Hz)

DMF
7-Amino-2-(4-fluorophenyl)-1,2,4-triazolo[1,5-a][1,3,5]triazine (5d)

7.41 (dd, J=8.76, 8.96 Hz, 1 H)
8.25 (dd, J=5.58, 8.78 Hz, 1 H)
Avance III - 13C DEPTQ-135 with IH Decoupling

116.00 (d, J=21.96 Hz, 2 C)
126.70 (d, J=2.94 Hz, 1 C)
129.30 (d, J=8.82 Hz, 2 C)
163.60 (d, J=247.94 Hz, 1 C)
7-Amino-2-(2-chlorophenyl)-1,2,4-triazolo[1,5-a][1,3,5]triazine (5e)

7.52 (dt, J=1.45, 7.43 Hz, 1 H)
7.57 (dt, J=1.93, 7.60 Hz, 1 H)
7.66 (dd, J=1.35, 7.85 Hz, 1 H)
7.93 (dd, J=1.88, 7.52 Hz, 1 H)
Avance III - 13C DEPTQ-135 with IH Decoupling
7-Amino-2-(3-chlorophenyl)-1,2,4-triazolo[1,5-a][1,3,5]triazine (5f)
7-Amino-2-(4-chlorophenyl)-1,2,4-triazolo[1,5-a][1,3,5]triazine (5g)

Current Data Parameters
NAME: AD-SK3
EXPNO: 1
PROC: 1

F2 - Acquisition Parameters
Date: 20120217
Time: 8.49
INSTRUM: spect
PROBHDL: 5 mm PABBO BB-
PULPN: 1g
TD: 48076
SOLVENT: DMSO
NS: 8
DS: 2
SMN: 6402.049 Hz
FIDRES: 0.133165 Hz
AQ: 3.7547855 sec
RG: 128
DM: 76.100 usec
DF: 45.02 usec
TR: 299.8 K
TE: 5.0000000 sec
DDT: 1

---------- CHANNEL f1 ----------
NUC1: 1H
P1: 14.50 usec
LIN1: 12.40300344 K
SFO1: 400.1326008 MHz

F2 - Processing parameters
SI: 32768
SF: 400.130030 MHz
NOW: EM
SSB: 0
LBK: 0.30 Hz
CA: n
FC: 1.00
13C DEPTQ-135 with IH Decoupling
7-Amino-2-(3-nitrophenyl)-1,2,4-triazolo[1,5-a][1,3,5]triazine (5h)

7.89 (t, J=8.02 Hz, 1 H)
8.40 (ddd, J=0.89, 2.33, 8.25 Hz, 1 H)
8.59 (td, J=1.26, 7.83 Hz, 1 H)
8.97 (t, J=1.84 Hz, 1 H)
7-Amino-2-(4-methylphenyl)-1,2,4-triazolo[1,5-a][1,3,5]triazine (5i)

N\textsubscript{N}\textsubscript{N}\textsubscript{N}NH\textsubscript{2}  

7.37 (d, J=8.1 Hz, 1 H)  
8.10 (d, J=8.1 Hz, 1 H)  

DMF

Current Data Parameters
NAME:  AD-3K8  
EXPNO:  1  
PROCNO:  1  

F2 - Acquisition Parameters
Date:  20120227  
Time:  5:23  
INSTRUM:  spect  
PROBND:  5 mm PABBO BB-  
PULP:  zg  
TD:  48076  
SOLVENT:  DMSO  
NS:  8  
DS:  2  
SWH:  6402.049 Hz  
FIDRES:  0.133165 Hz  
AQ:  3.7547855 sec  
RG:  161  
DM:  78.100 usec  
DE:  15.52 usec  
TE:  299.6 K  
DI:  5.00000000 sec  
TD0:  1  

---------- CHANNEL F1 ----------
NUC1:  1H  
P1:  14.50 usec  
PLW1:  12.42300034 M  
SP01:  400.1330010 MHz  

F2 - Processing parameters
SI:  32768  
SP:  400.1330017 MHz  
WDM:  RM  
SSB:  0  
LB:  0.30 Hz  
GB:  0  
P:  1.00
7-Amino-2-(3-methoxyphenyl)-1,2,4-triazolo[1,5-a][1,3,5]triazine (5j)

N N
NH2
MeO

7.12 (ddd, J=0.96, 2.68, 8.38 Hz, 1 H)
7.48 (dd, J=7.70, 8.37 Hz, 1 H)
7.74 (ddd, J=1.46, 2.62 Hz, 1 H)
7.81 (ddd, J=1.02, 1.38, 7.70 Hz, 1 H)
7-Amino-2-(4-trifluoromethylphenyl)-1,2,4-triazolo[1,5-a][1,3,5]triazine (5k)

F₃C

NH₂

N
N
N

8.41 (d, J=8.2 Hz, 2 H)
7.96 (d, J=8.2 Hz, 2 H)

Current Data Parameters
NAME    SK28
EXPNDO  2
PROCNO  1

F2 - Acquisition Parameters
Date_    20120423
Time_    9.28
INSTRUM  spect
PROBHD   5 mm PABBO BB-
PULPROG   XG
TD       48076
SOLVENT  DMSC
NS       8
DS       2
SWH      6602.049 Hz
FIDRES   0.133165 Hz
AQ       3.7567955 sec
RG       114
DW       78.100 usec
DE       15.02 usec
TE       299.8 K
D1       5.0000000 sec
T10       1

F2 - Processing parameters
SI       32768
SF       400.1299946 MHz
KOW      EM
SSB      0
LB       0.30 Hz
GB       0
PC       1.00
7-Amino-2-(2-furyl)-1,2,4-triazolo[1,5-a][1,3,5]triazine (5I)

![Chemical structure of 7-Amino-2-(2-furyl)-1,2,4-triazolo[1,5-a][1,3,5]triazine (5I)]

- **6.74 (dd, J=1.76, 3.40 Hz, 1 H)**
- **7.23 (dd, J=0.80, 3.40 Hz, 1 H)**
- **7.95 (dd, J=0.78, 1.74 Hz, 1 H)**
13C DEPT-135 with IH Decoupling
7-Amino-2-(2-thienyl)-1,2,4-triazolo[1,5-a][1,3,5]triazine (5m)

![Chemical structure](image)

**NMR Spectra**

- **Chemical Shifts**
  - 7.25 (dd, J=3.64, 5.00 Hz, 1 H)
  - 7.80 (dd, J=1.22, 5.02 Hz, 1 H)
  - 7.85 (dd, J=1.24, 3.64 Hz, 1 H)

**Current Data Parameters**
- **NAME**: AD-359
- **FPROCNO**: 1

**F1: Acquisition Parameters**
- **Date**: 20120305
- **Time**: 8.36
- **INSTRUM**: spect
- **PROBHD**: 5 mm PABBO BB-
- **PULPROG**: zg
- **TD**: 48076
- **SOLVENT**: DMSO
- **NS**: 8
- **DS**: 2
- **SW1**: 6402.049 Hz
- **FIDRES**: 0.133165 Hz
- **AQ**: 3.7547855 sec
- **RG**: 144
- **DM**: 78.100 usec
- **DE**: 15.52 usec
- **TE**: 299.8 K
- **D1**: 5.0000000 sec
- **TD0**: 1

**F2: Processing parameters**
- **SI**: 32768
- **SF**: 400.129997 MHz
- **MDW**: EM
- **SSB**: 0
- **LB**: 0.30 Hz
- **GB**: 0
- **PC**: 1.00
Avance III - 13C DEPTQ-135 with 1H Decoupling
7-Amino-2-(3-pyridyl)-1,2,4-triazolo[1,5-a][1,3,5]triazine (5n)

[Chemical structure image]

Electronic Supplementary Material (ESI) for RSC Advances
This journal is © The Royal Society of Chemistry 2013
Avance III – 13C DEPTQ-135 with IH Decoupling
7-Amino-2-(4-pyridyl)-1,2,4-triazolo[1,5-a][1,3,5]triazine (50)

8.10 (dd, J=1.62, 4.42 Hz, 2 H)
8.80 (dd, J=1.62, 4.42 Hz, 2 H)