

1-Aminotetrazol-5-one and its salts : a family of new insensitive nitrogen rich energetic compounds

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and Jin-Ting Wu^[a]

Supporting Information

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NMR, MS and IR Spectra for all energetic compounds

¹³C NMR

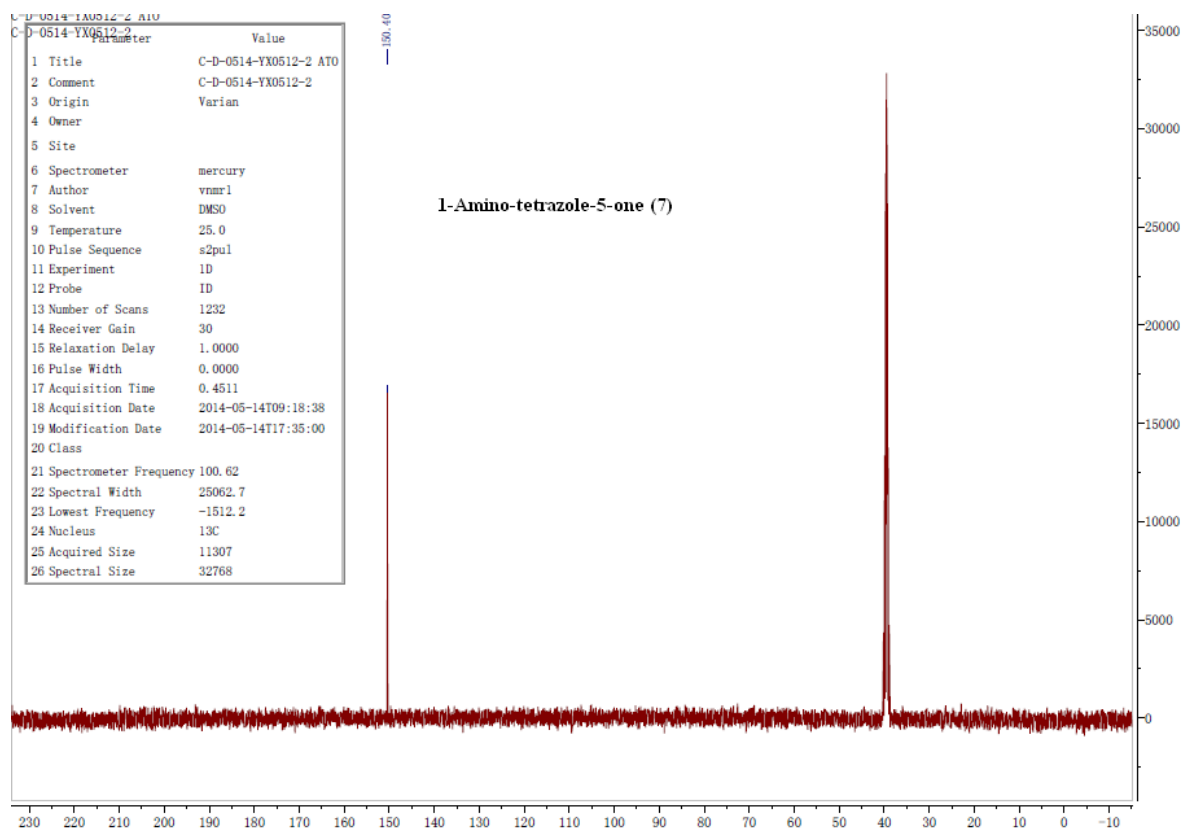


Figure S1. ¹³C NMR of 7

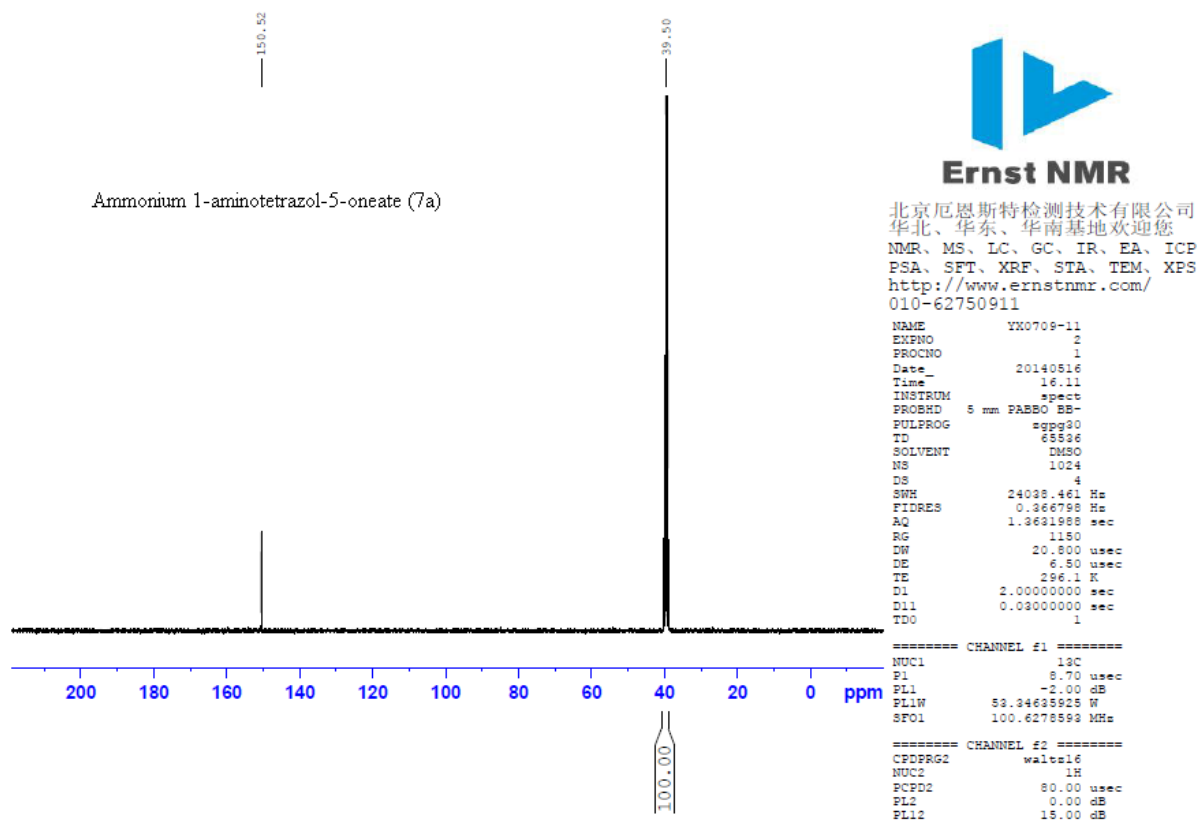


Figure S2. ¹³C NMR of 7a

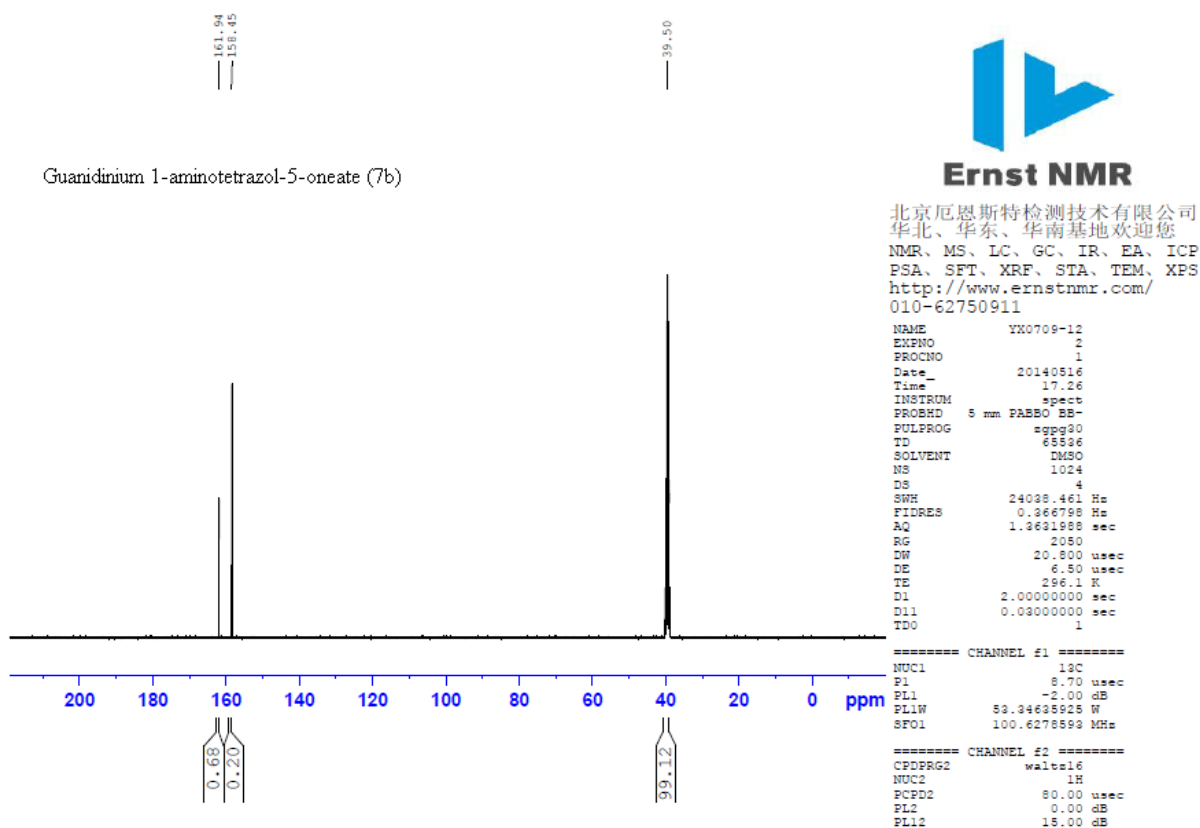


Figure S3. ¹³C NMR of 7b

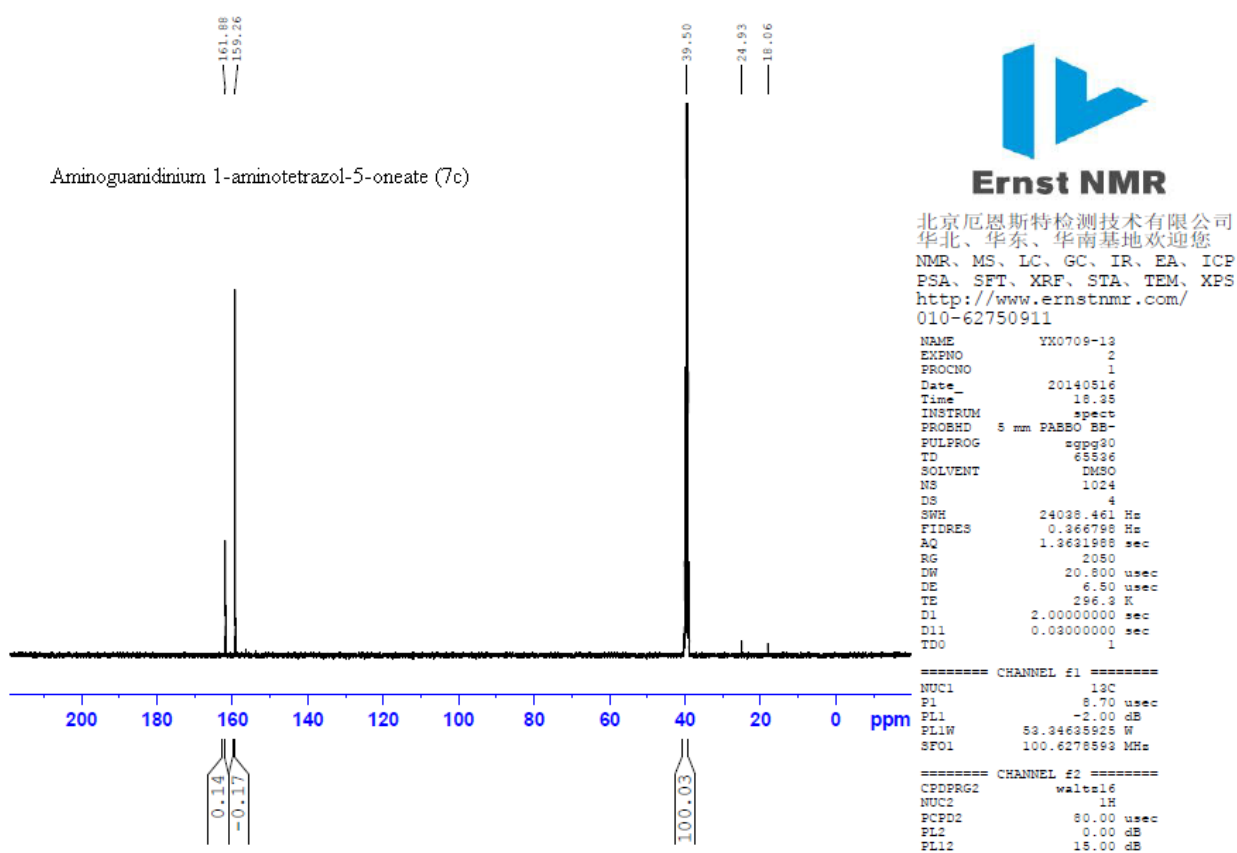
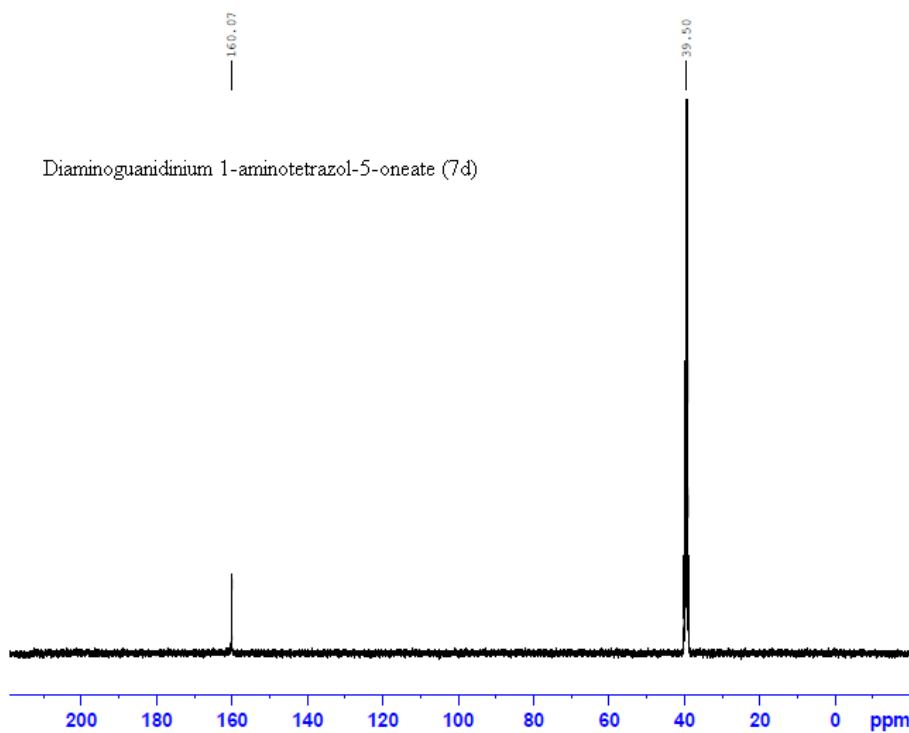


Figure S4. ¹³C NMR of 7c



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 010-62750911

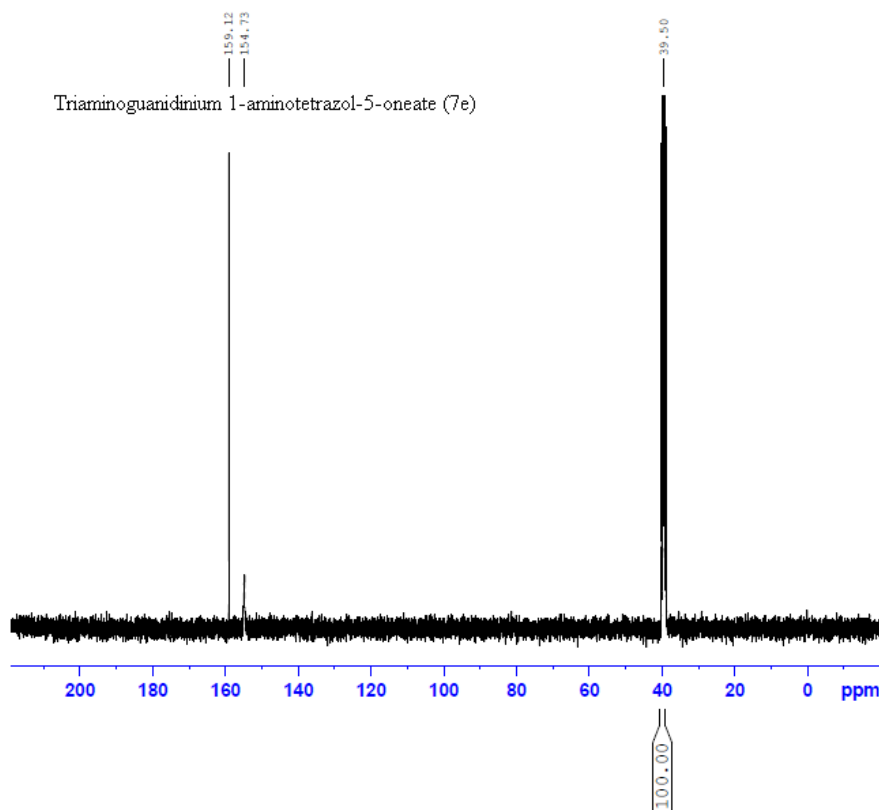
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PROCNO        1
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TD            65536
SOLVENT       DMSO
NS            1024
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG            2050
DW            20.800 usec
DE            6.50 usec
TE            298.6 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          13C
P1            8.70 usec
PL1           -2.00 dB
PL1W          53.34635925 W
SFO1          100.6278593 MHz

===== CHANNEL f2 =====
CPDPRG2      waltz16
NUC2          1H
PCPD2        80.00 usec
PL2           0.00 dB
PL12          15.00 dB
  
```

Figure S5. ^{13}C NMR of 7d



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NS            1024
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG            2050
DW            20.800 usec
DE            6.50 usec
TE            298.6 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          13C
P1            8.70 usec
PL1           -2.00 dB
PL1W          53.34635925 W
SFO1          100.6278593 MHz

===== CHANNEL f2 =====
CPDPRG2      waltz16
NUC2          1H
PCPD2        80.00 usec
PL2           0.00 dB
PL12          15.00 dB
  
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Figure S6. ^{13}C NMR of 7e

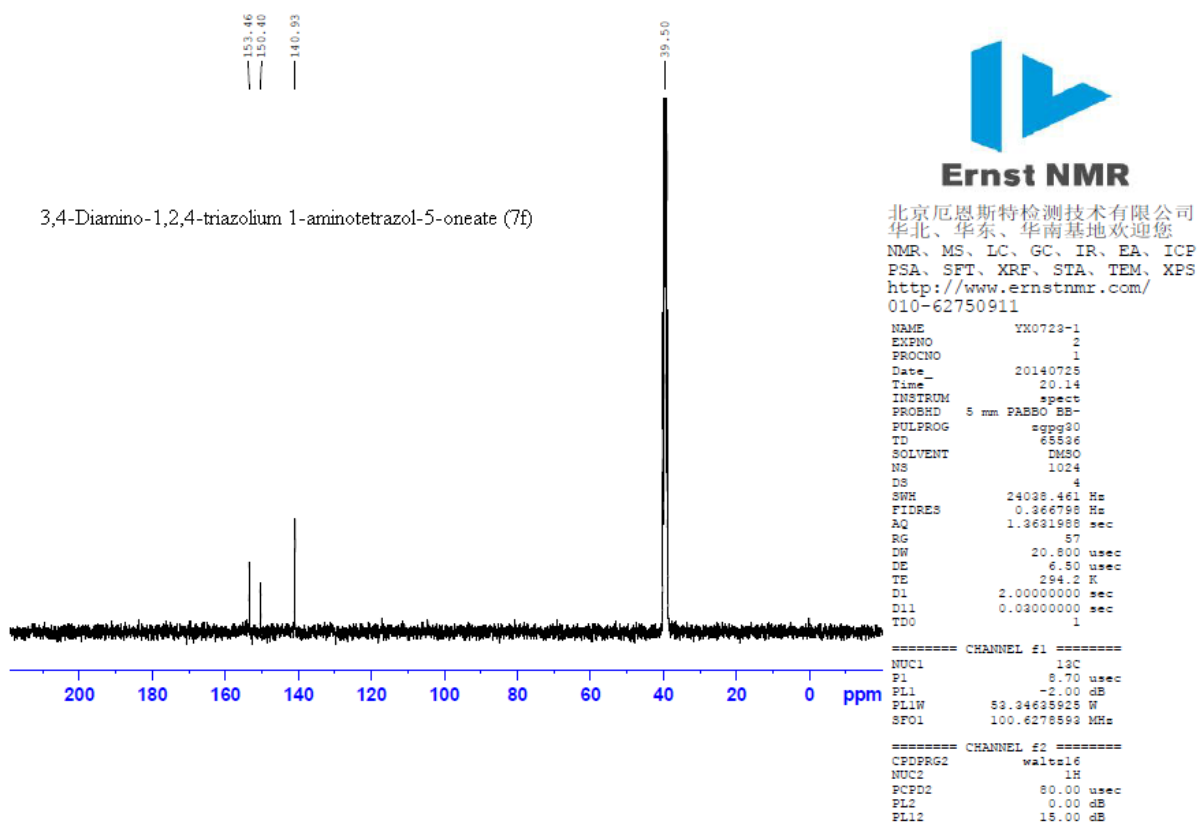


Figure S7. ^{13}C NMR of 7f

MS

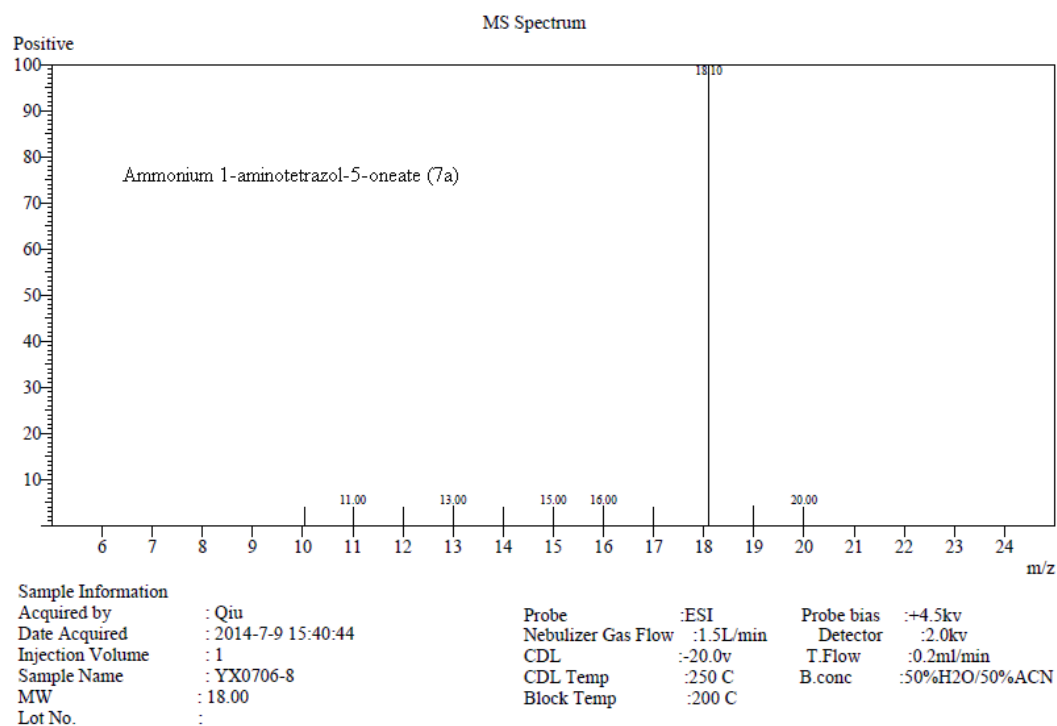


Figure S8. MS(ESI⁺) of 7a

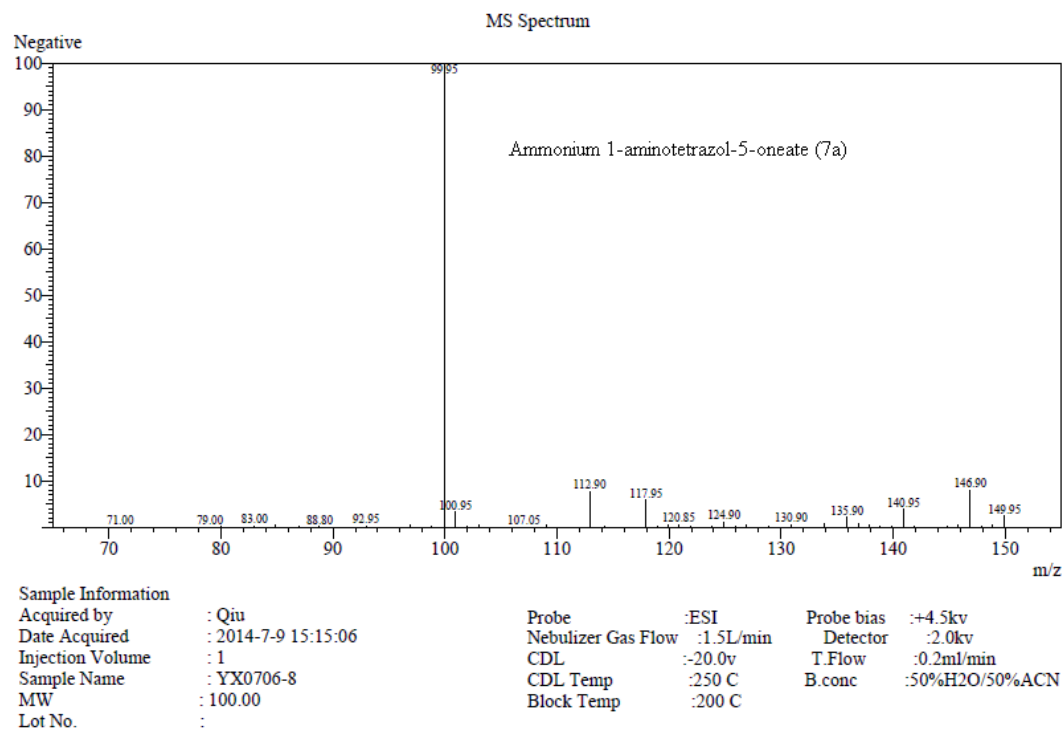


Figure S9. MS(ESI⁺) of 7a

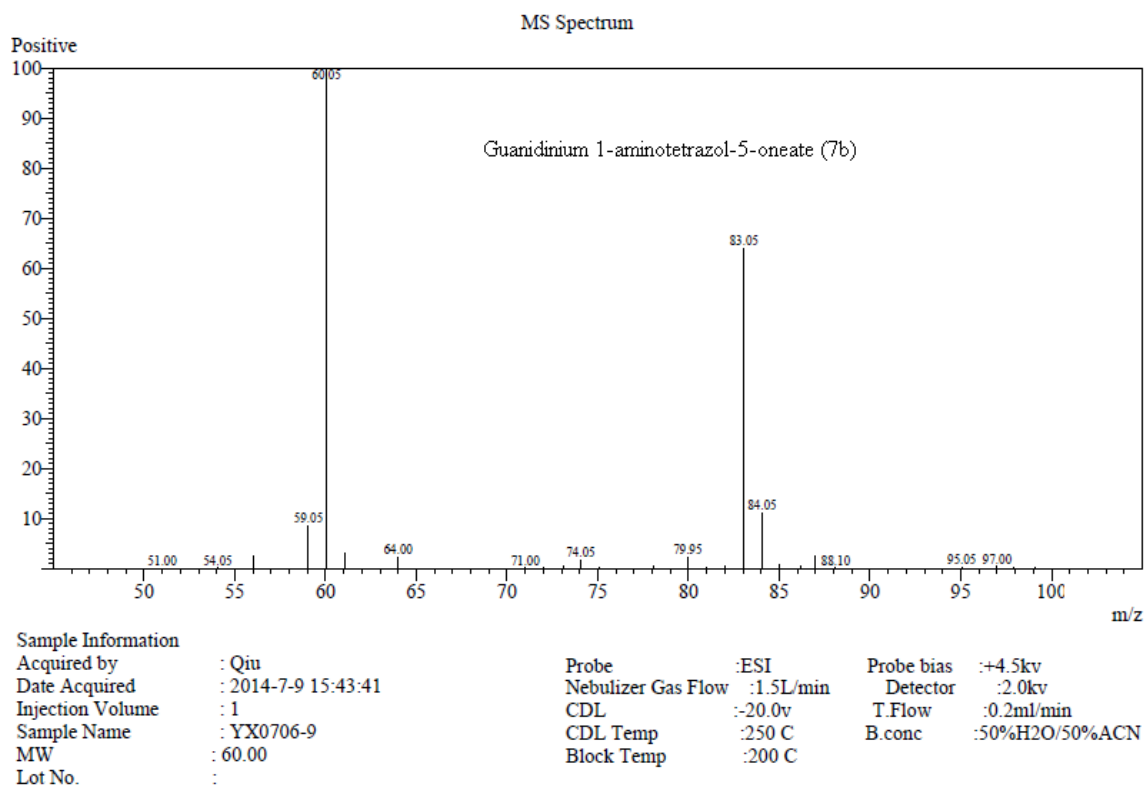


Figure S10. MS(ESI⁺) of 7b

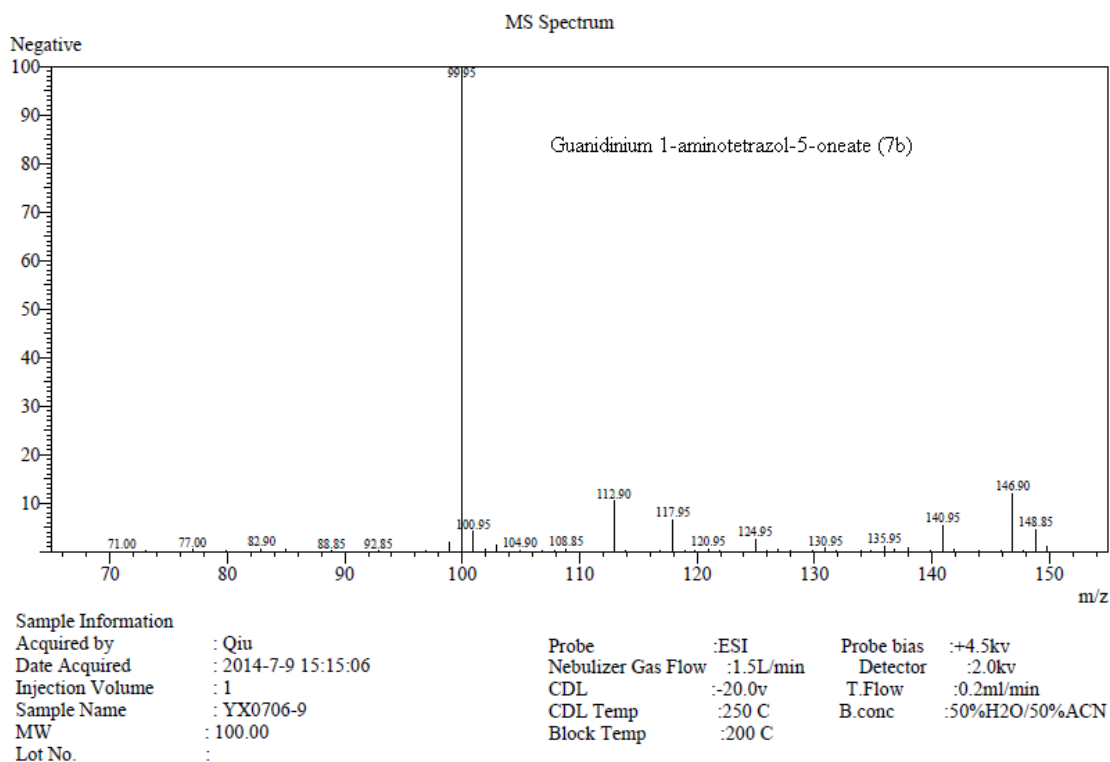


Figure S11. MS(ESI⁻) of 7b

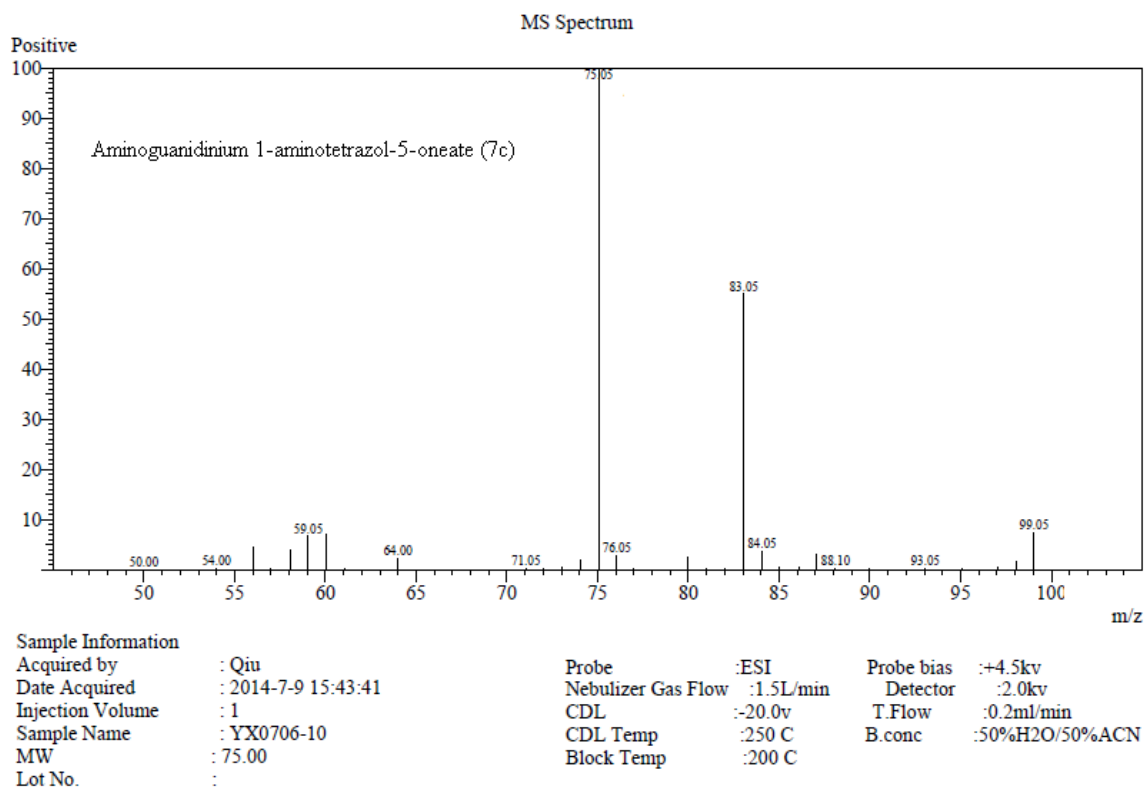


Figure S12. MS(ESI⁺) of 7c

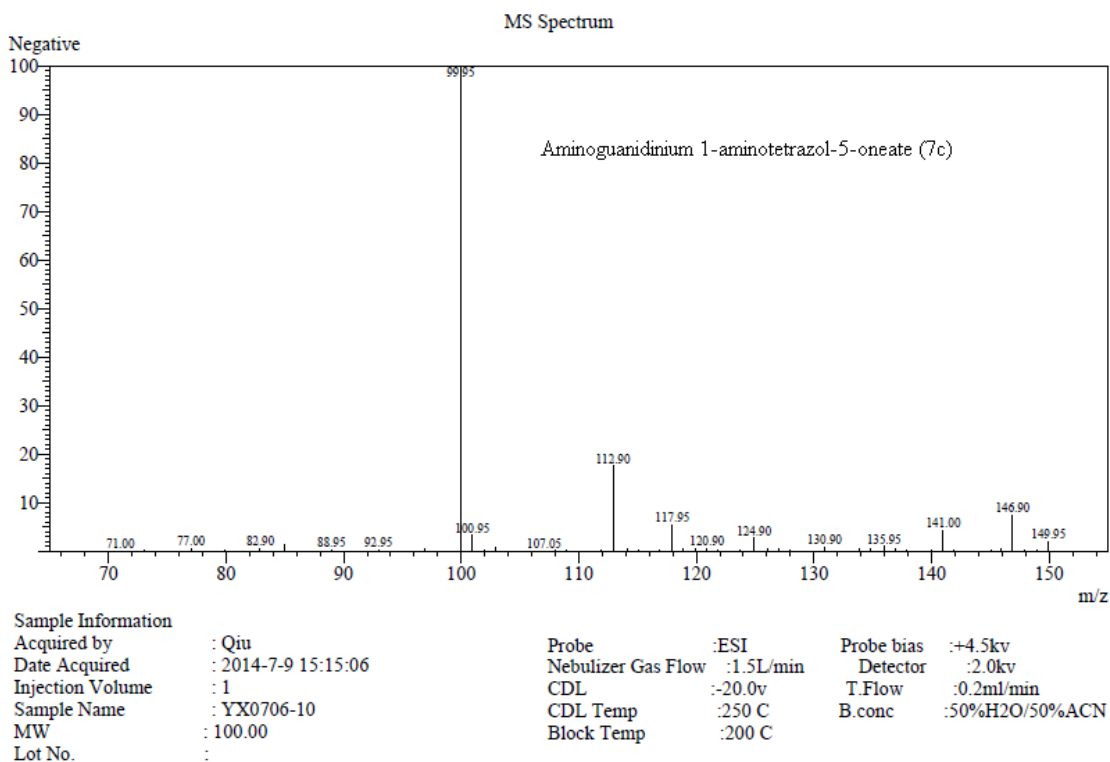


Figure S13. MS(ESI⁻) of 7c

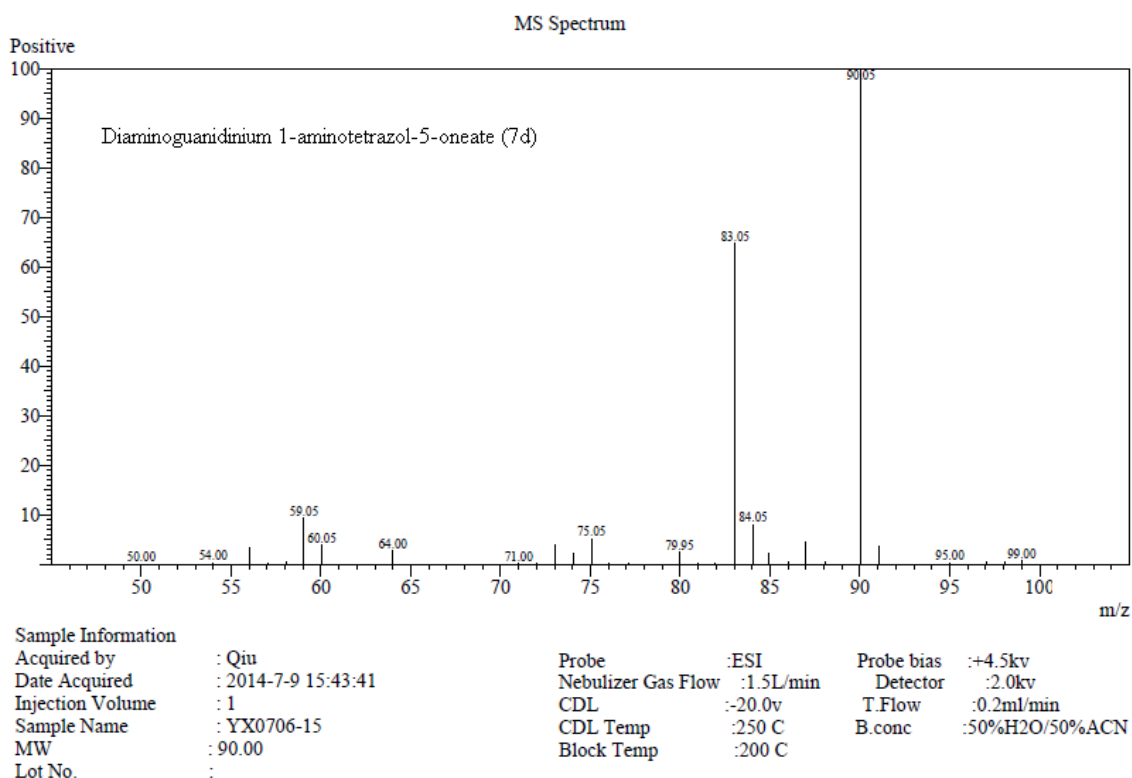
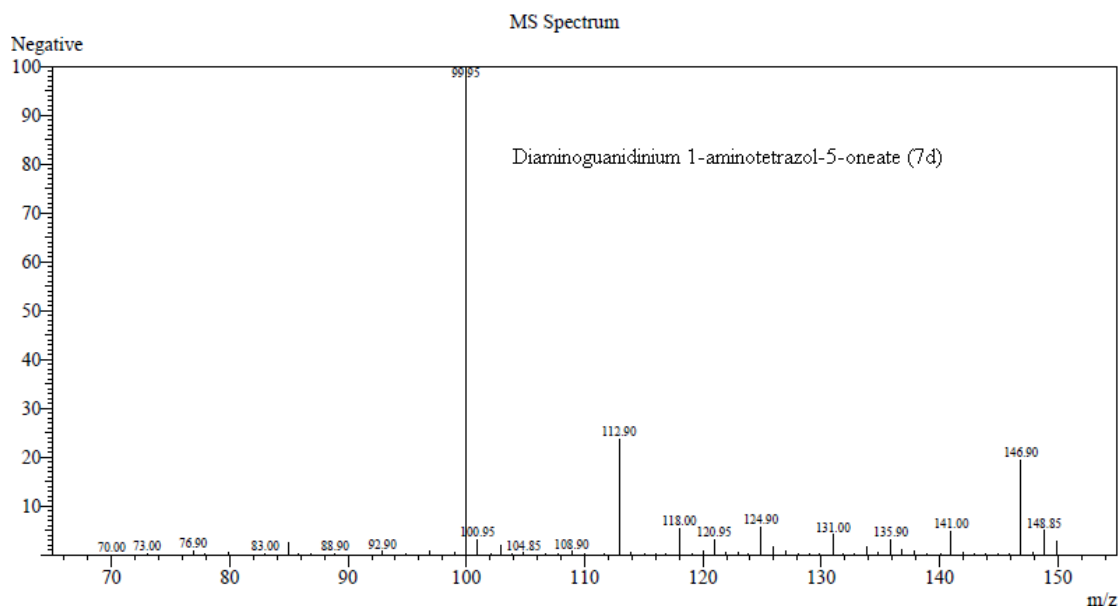


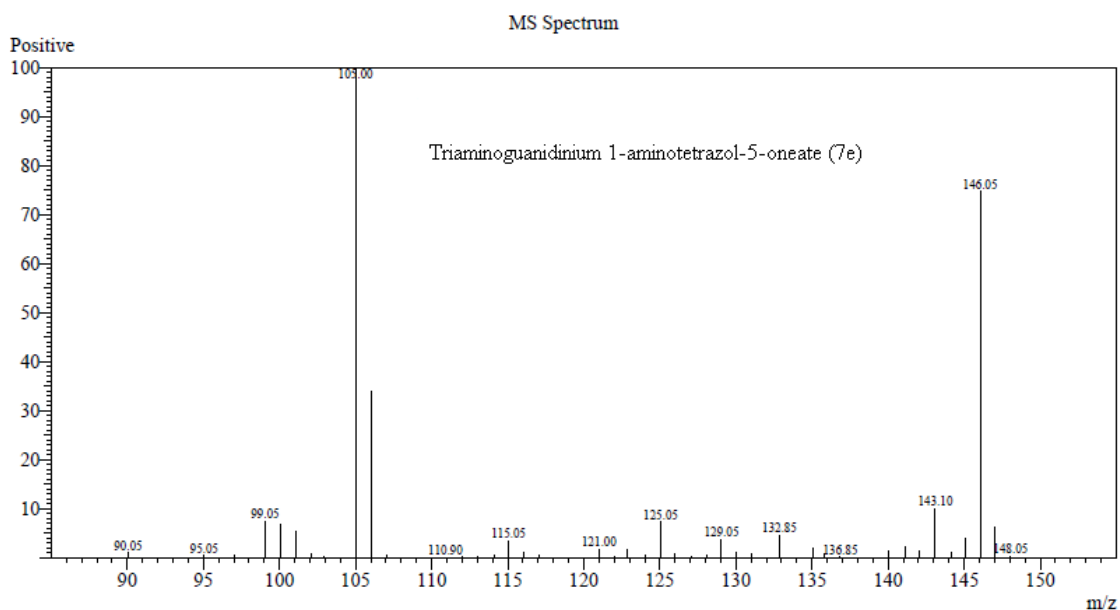
Figure S14. MS(ESI⁺) of 7d



Sample Information

Acquired by	: Qiu	Probe	: ESI	Probe bias	: +4.5kv
Date Acquired	: 2014-7-9 15:15:06	Nebulizer Gas Flow	: 1.5L/min	Detector	: 2.0kv
Injection Volume	: 1	CDL	: -20.0v	T.Flow	: 0.2ml/min
Sample Name	: YX0706-15	CDL Temp	: 250 C	B.conc	: 50%H2O/50%ACN
MW	: 100.00	Block Temp	: 200 C		
Lot No.	:				

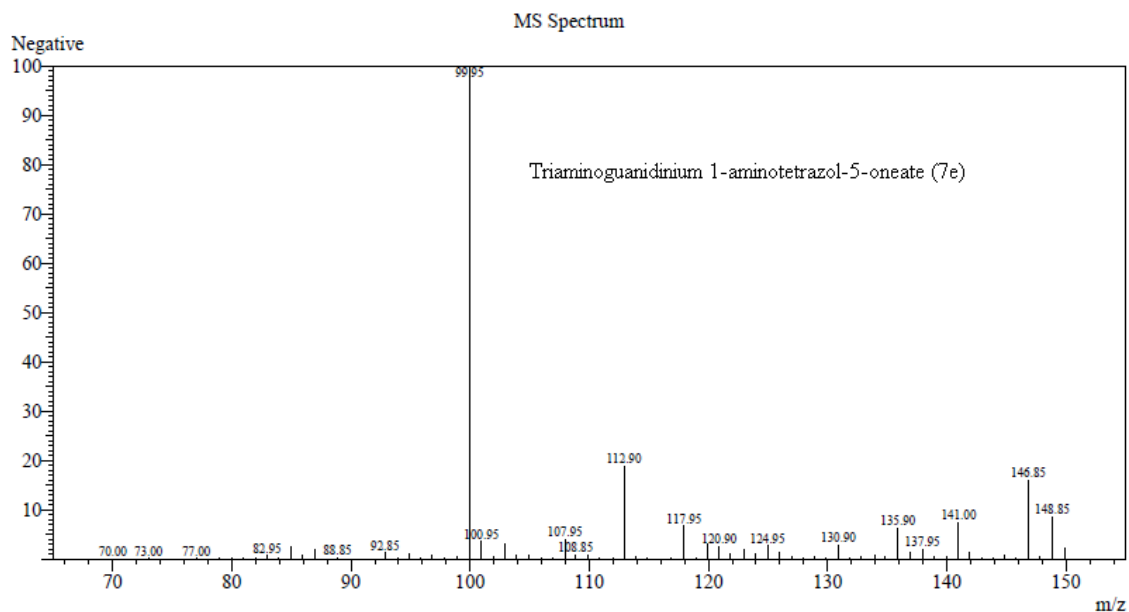
Figure S15. MS(ESI⁻) of 7d



Sample Information

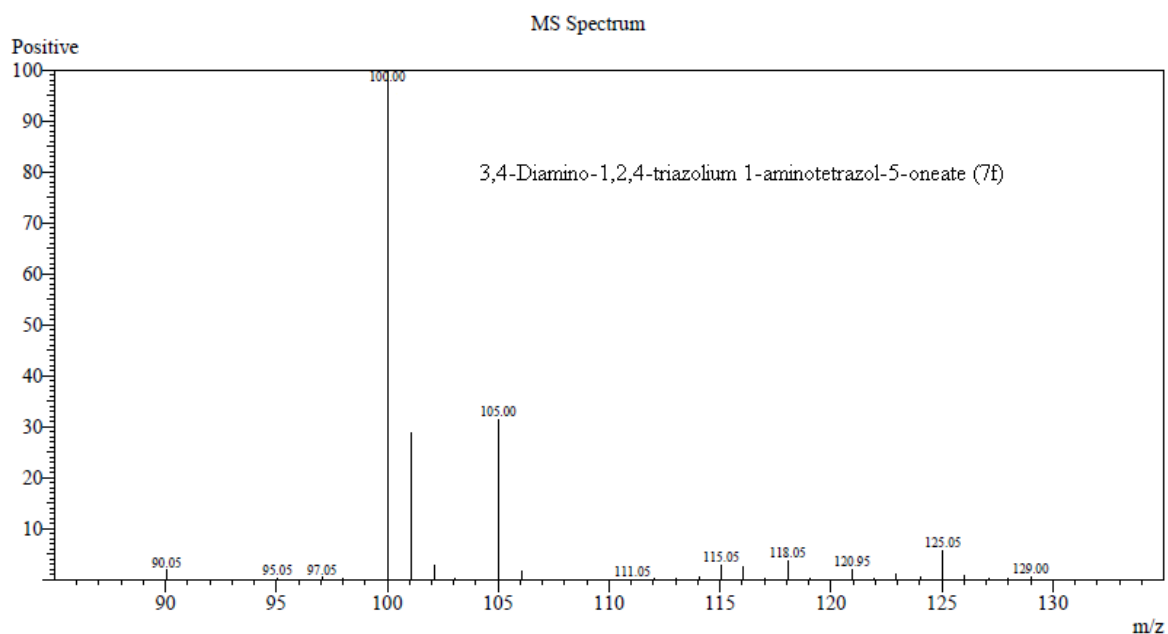
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Injection Volume	: 1	CDL	: -20.0v	T.Flow	: 0.2ml/min
Sample Name	: YX0706-14	CDL Temp	: 250 C	B.conc	: 50%H2O/50%ACN
MW	: 105.00	Block Temp	: 200 C		
Lot No.	:				

Figure S16. MS(ESI⁺) of 7e



Sample Information			
Acquired by	: Qiu	Probe	: ESI
Date Acquired	: 2014-7-9 15:15:06	Probe bias	: +4.5kv
Injection Volume	: 1	Nebulizer Gas Flow	: 1.5L/min
Sample Name	: YX0706-14	Detector	: 2.0kv
MW	: 100.00	CDL	: -20.0v
Lot No.	:	T.Flow	: 0.2ml/min
		CDL Temp	: 250 C
		Block Temp	: 200 C
		B.conc	: 50%H2O/50%ACN

Figure S17. MS(ESI⁻) of 7e



Sample Information			
Acquired by	: Qiu	Probe	: ESI
Date Acquired	: 2014-7-9 15:52:34	Probe bias	: +4.5kv
Injection Volume	: 1	Nebulizer Gas Flow	: 1.5L/min
Sample Name	: WJT0704-2	Detector	: 2.0kv
MW	: 100.00	CDL	: -20.0v
Lot No.	:	T.Flow	: 0.2ml/min
		CDL Temp	: 250 C
		Block Temp	: 200 C
		B.conc	: 50%H2O/50%ACN

Figure S18. MS(ESI⁺) of 7f

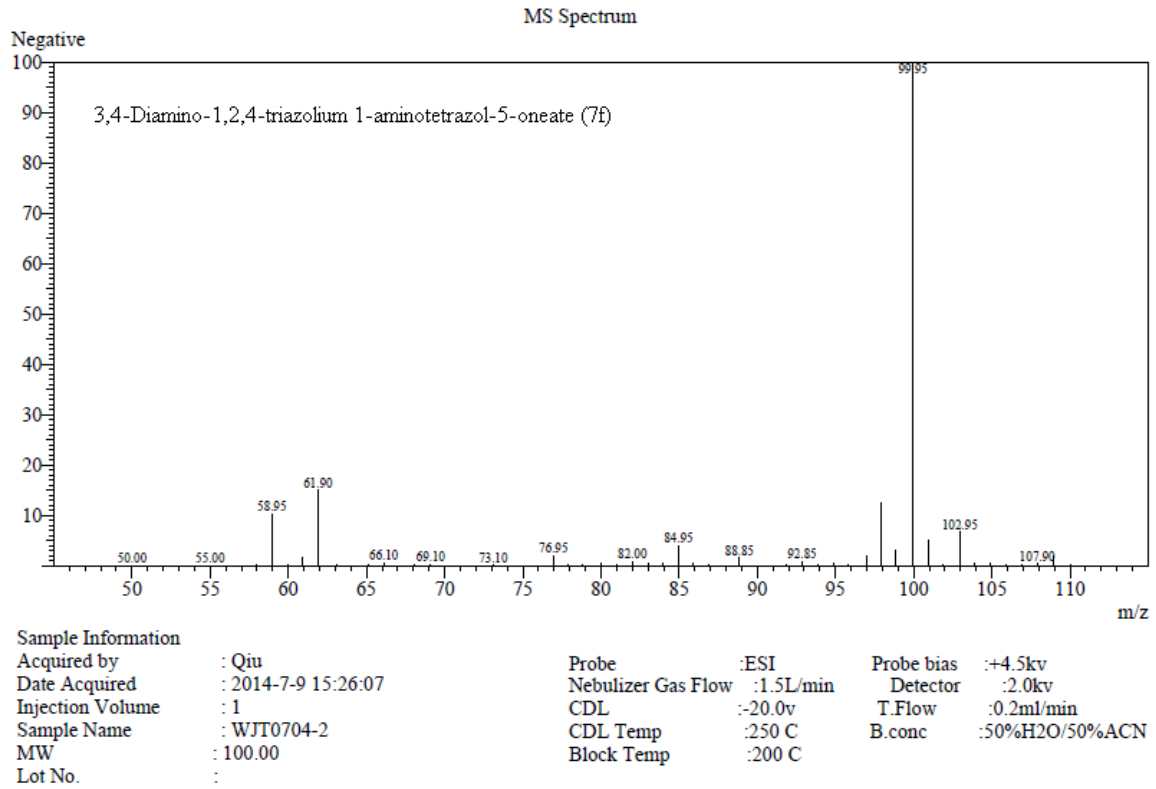


Figure S19. MS(ESI) of 7f

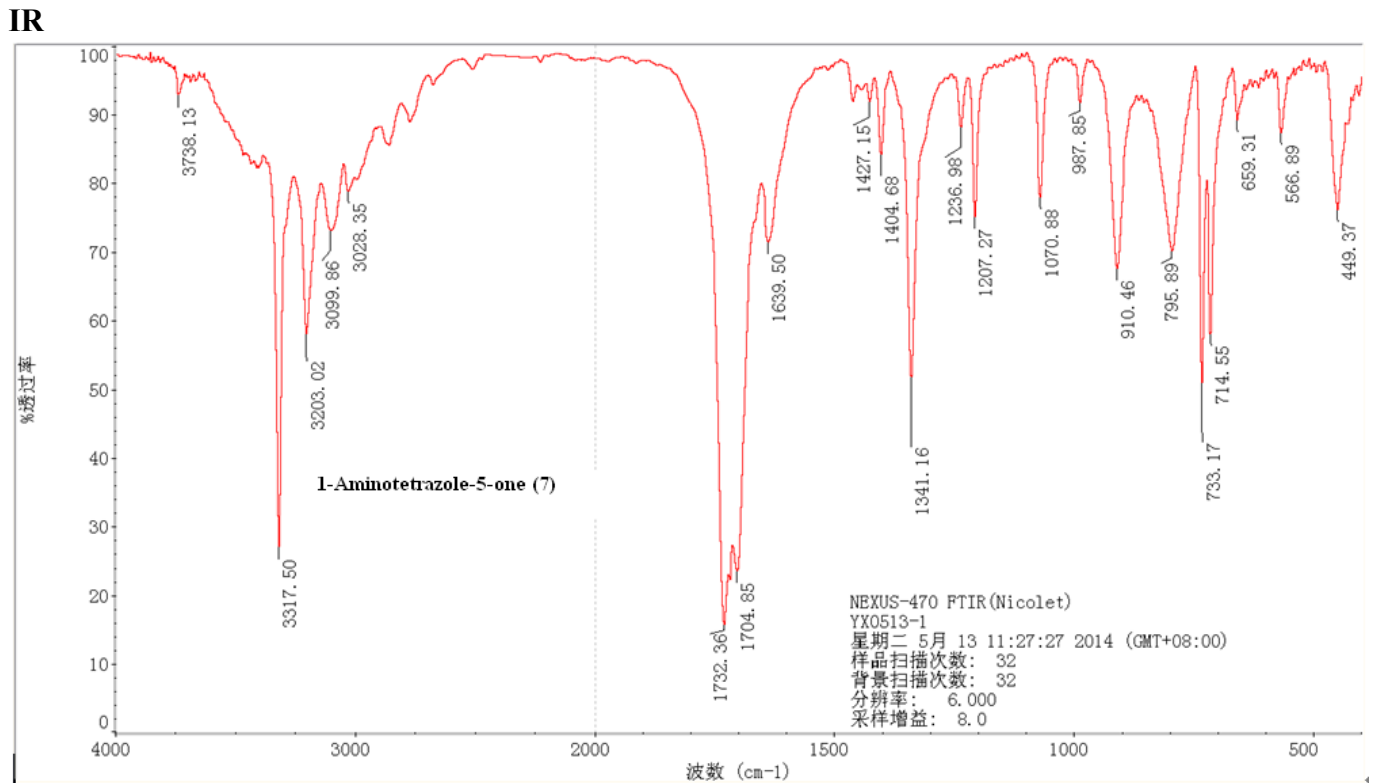


Figure S20. IR of 7

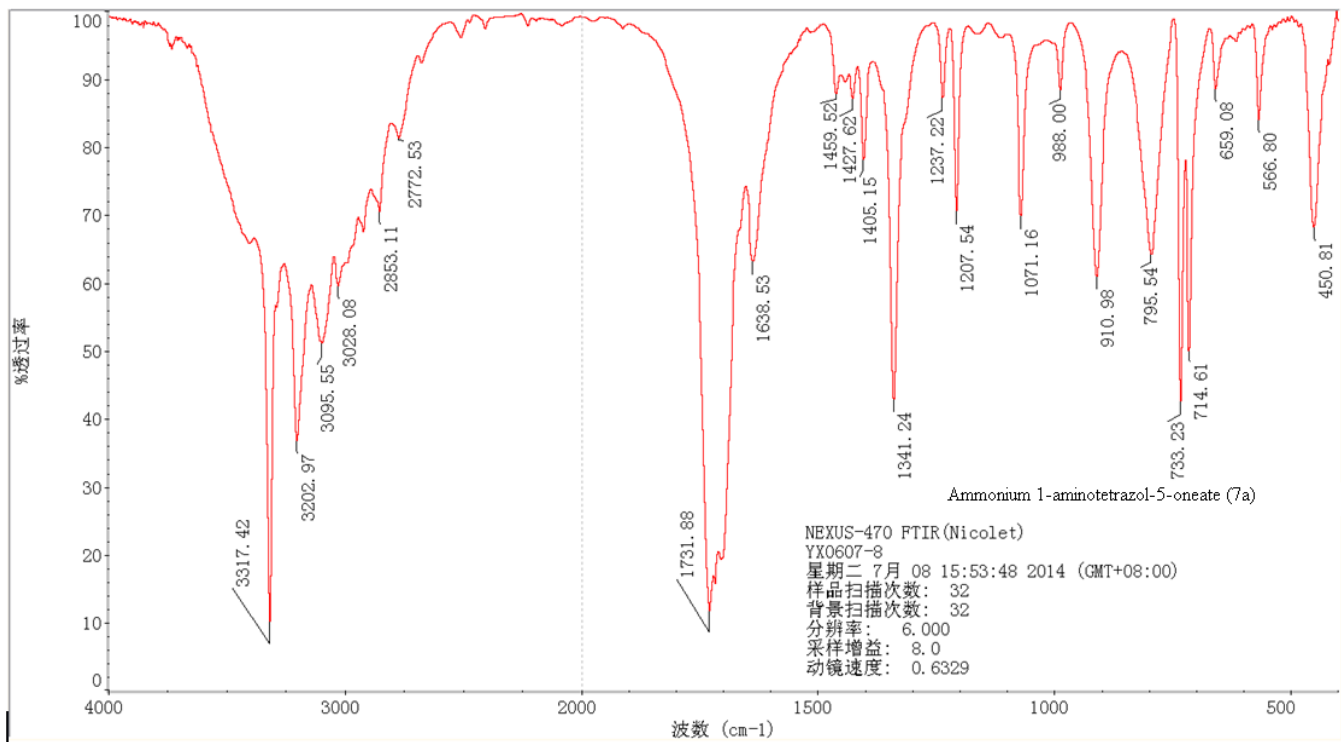


Figure S21. IR of 7a

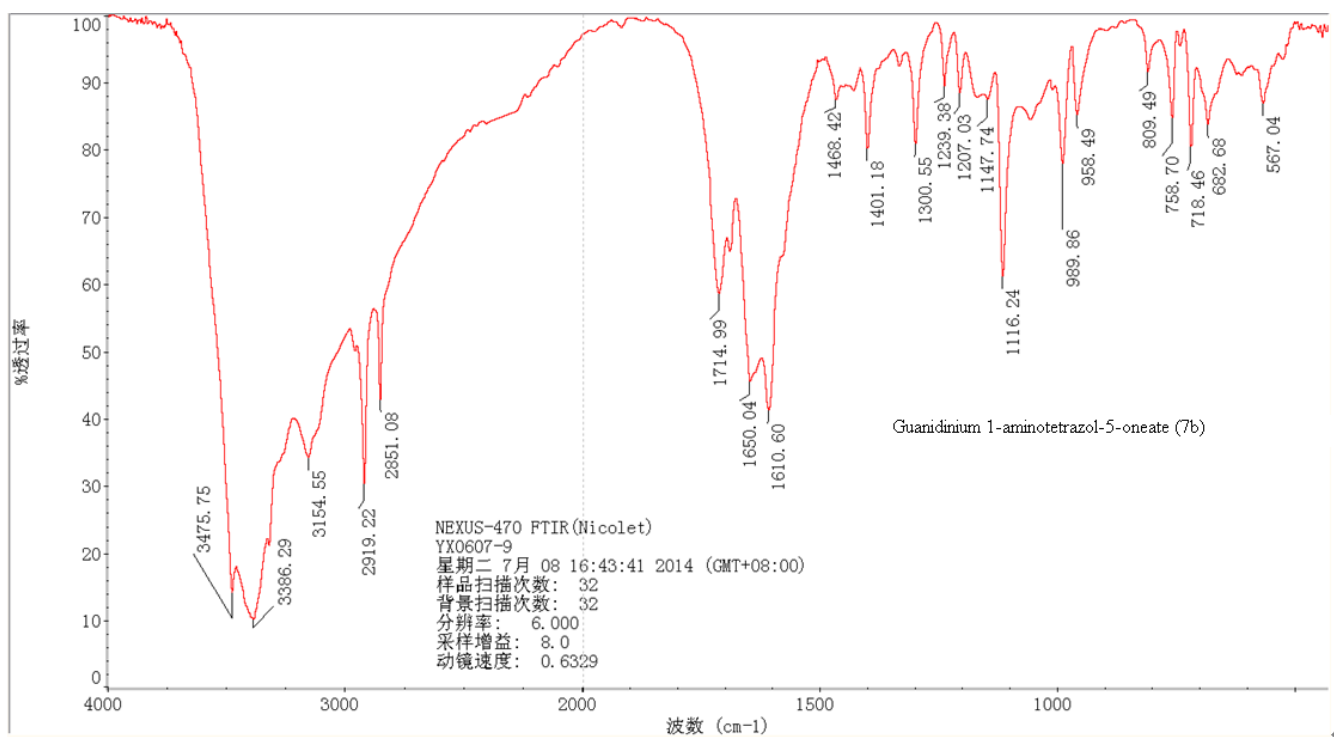


Figure S22. IR of 7b

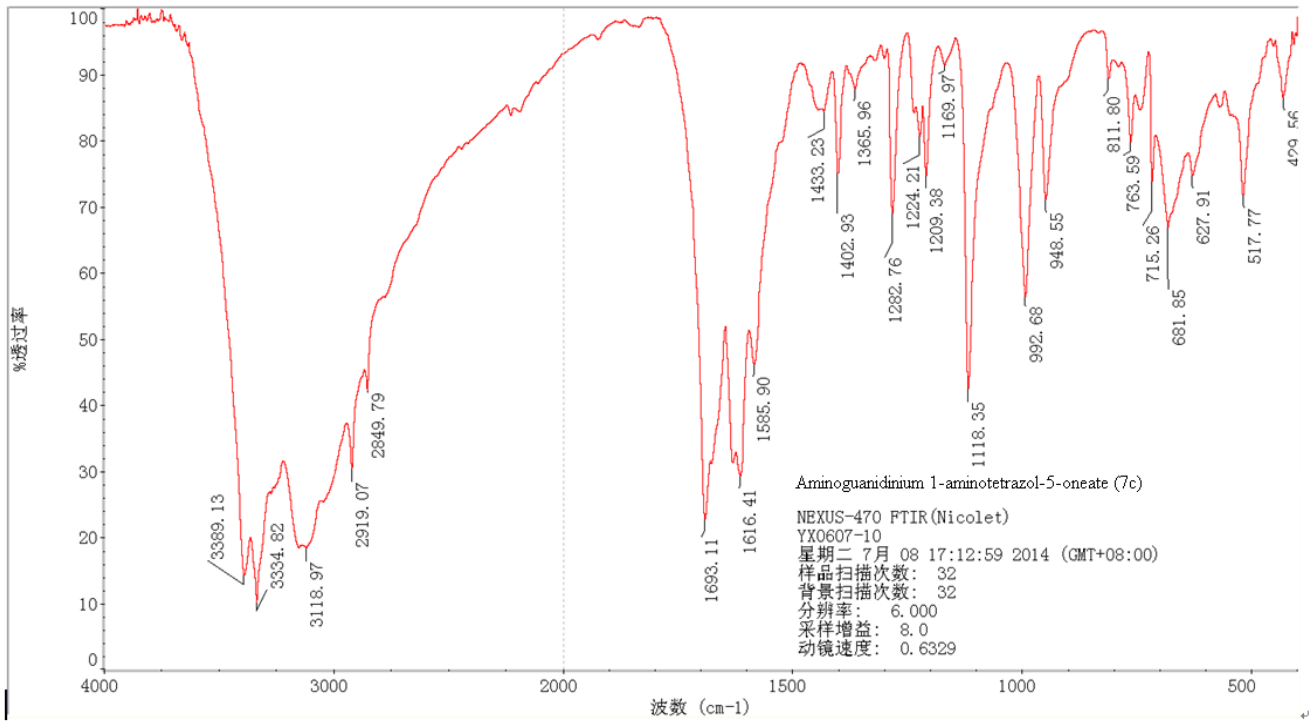


Figure S23. IR of 7c

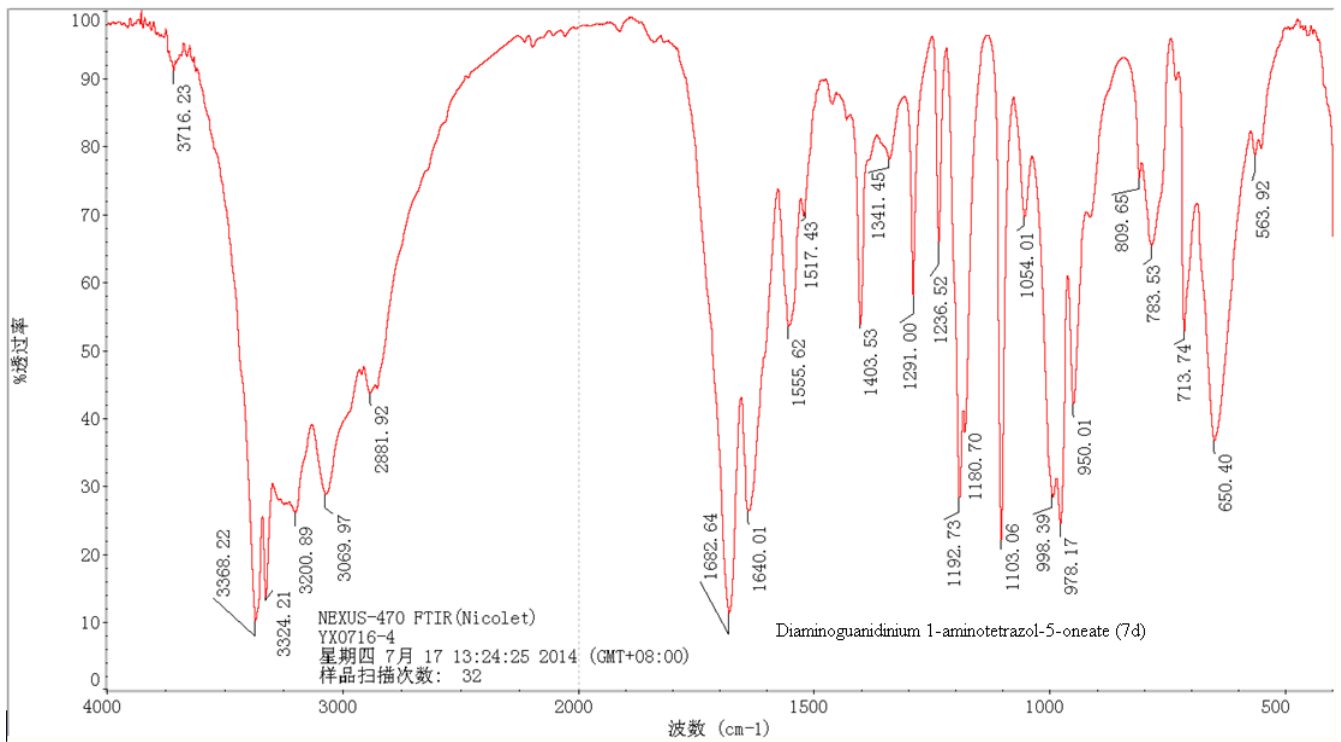


Figure S24. IR of 7d

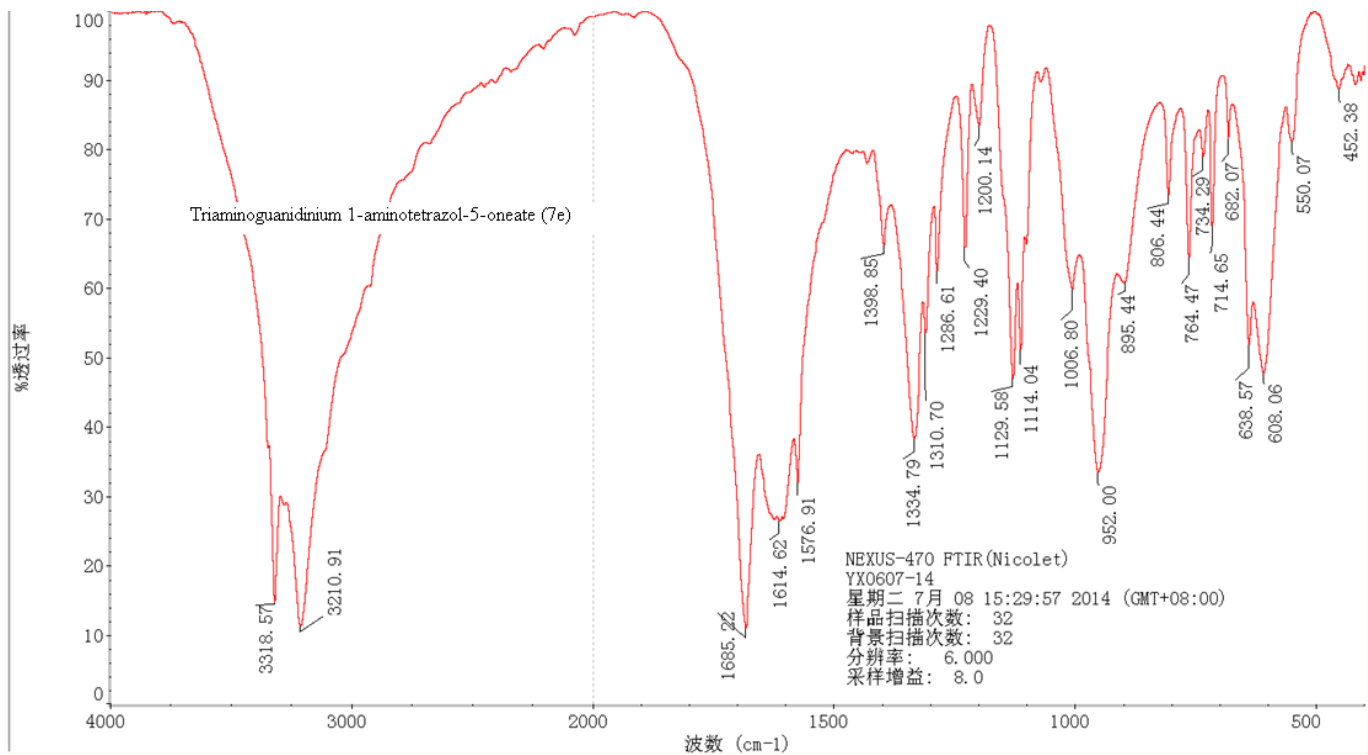


Figure S25. IR of 7e

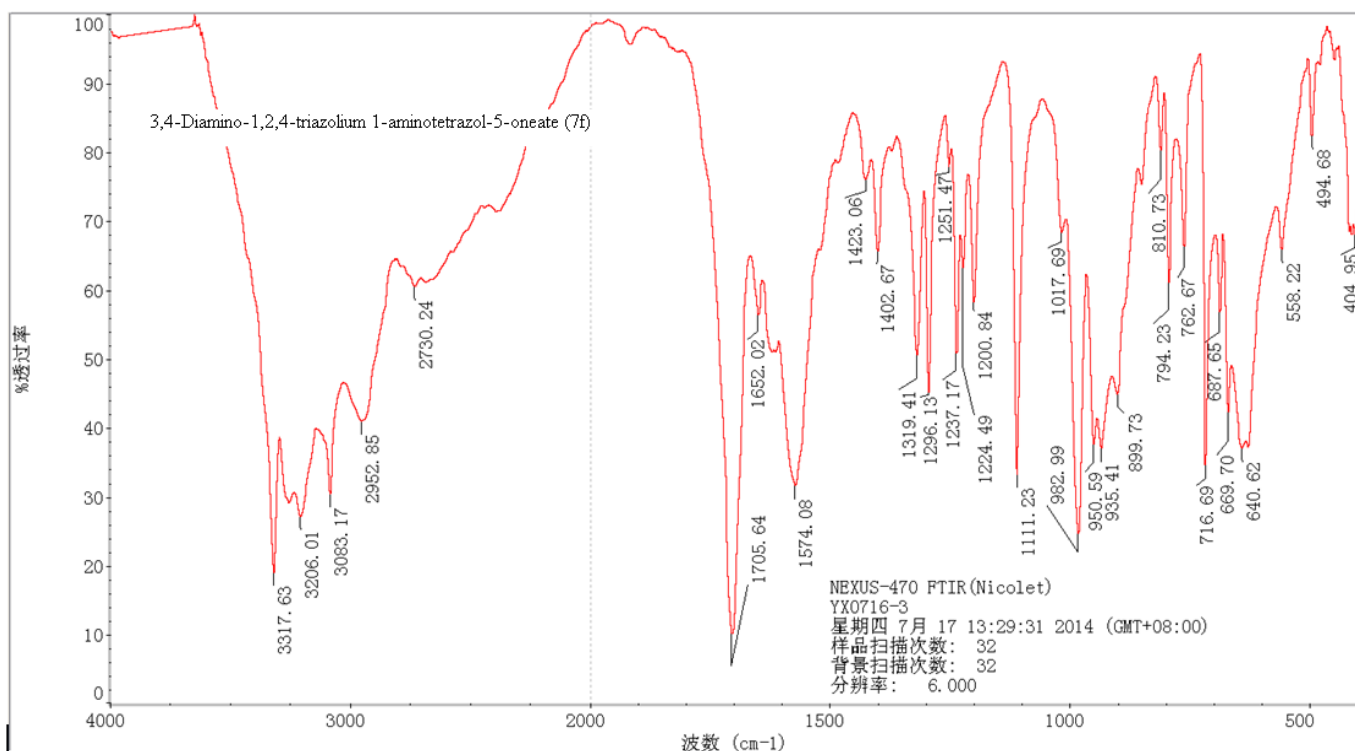


Figure S26. IR of 7f

Crystallographic data

Table S1. Crystallographic data for 1-amino-tetrazol-5-one and its salts

	7	7a	7b	7c	7d	7e	7f
Formula	CH ₃ N ₅ O	CH ₆ N ₆ O	C ₂ H ₈ N ₈ O	C ₂ H ₉ N ₉ O	C ₂ H ₁₀ N ₁₀ O	C ₂ H ₁₁ N ₁₁ O	C ₃ H ₈ N ₁₀ O
FW /g·mol ⁻¹	101.08	118.12	160.16	175.18	190.20	205.22	200.19
Crystal system	orthorhombic	monoclinic	monoclinic	monoclinic	monoclinic	orthorhombic	triclinic
Space group	Pbca	P2(1)/c	C2/c	P2(1)/n	P2(1)/n	Pbca	P-1
Color/habit	white/block	white/block	white/block	yellow/block	yellow/block	yellow/block	white/block
Size/mm	0.37×0.35× 0.16	0.36×0.15× 0.12	0.43×0.42× 0.32	0.34×0.22× 0.17	0.21×0.13× 0.06	0.20×0.20× 0.20	0.15×0.12× 0.07
<i>a</i> /Å	9.8873(7)	10.6535(7)	12.7785(11)	8.5361(4)	7.0441(5)	12.8237(6)	6.0700(6)
<i>b</i> /Å	5.5314(3)	6.5711(5)	6.4100(6)	6.5988(3)	12.0324(11)	6.7049(3)	7.1682(7)
<i>c</i> /Å	13.6734(9)	6.9431(6)	16.5787(15)	13.2935(7)	10.1093(8)	20.2018(10)	10.6018(9)
<i>α</i> /°	90.00	90.00	90.00	90.00	90.00	90.00	76.8520(10)
<i>β</i> /°	90.00	101.531(2)	93.3030(10)	103.3740(10)	105.964(2)	90.00	88.343(2)
<i>γ</i> /°	90.00	90.00	90.00	90.00	90.00	90.00	65.4060(10)
<i>V</i> /Å ³	747.81(8)	476.24(6)	1355.7(2)	728.49(6)	823.79(12)	1736.98(14)	407.32(7)
<i>Z</i>	8	4	8	4	4	8	2
$\rho_{\text{calcd.}}$ /g·cm ⁻³	1.796	1.647	1.569	1.597	1.534	1.569	1.632
μ /mm ⁻¹	0.154	0.139	0.128	0.130	0.125	0.128	0.132
<i>F</i> (000)	416	248	672	368	400	864	208
<i>T</i> /K	298	298	298	296	298	296	298
θ Min-max /°	2.98, 28.61	3.672, 23.065	2.461, 27.731	2.59, 28.68	2.69, 25.00	2.02, 28.57	3.22, 25.01
Dataset h; k; l	-11:13; -7:7; - 14:18	-12:12; -7:7; - 8:8	-15:13; -7:5; - 19:19	-11:10; -6:8; - 17:17	-8:8; -10:14; - 12:10	-17:15; -6:8; - 26:26	-7:5; -8:8; - 12:11
Reflections collected	4355	838	3139	5898	4077	13549	2105
Independent refl.	954	838	1201	1862	1452	2195	1427
<i>R</i> _{int}	0.0557	0.0000	0.0329	0.0162	0.0758	0.0362	0.0513
Parameters	64	74	100	129	118	171	127
<i>R</i> ₁ (obs)	0.0761	0.0965	0.0602	0.0445	0.0836	0.0533	0.1121
w <i>R</i> ₂ (all data)	0.1140	0.2048	0.1169	0.1092	0.0908	0.0985	0.2065
Resd. dens. /e·Å ⁻³	0.233, -0.218	0.352, -0.376	0.154, -0.213	0.205, -0.219	0.206, -0.230	0.164, -0.255	0.470, -0.450
CCDC	1018898	1018899	1018904	1018893	1018919	1018921	1018905

Packing diagram and Hydrogen bond for all energetic compounds

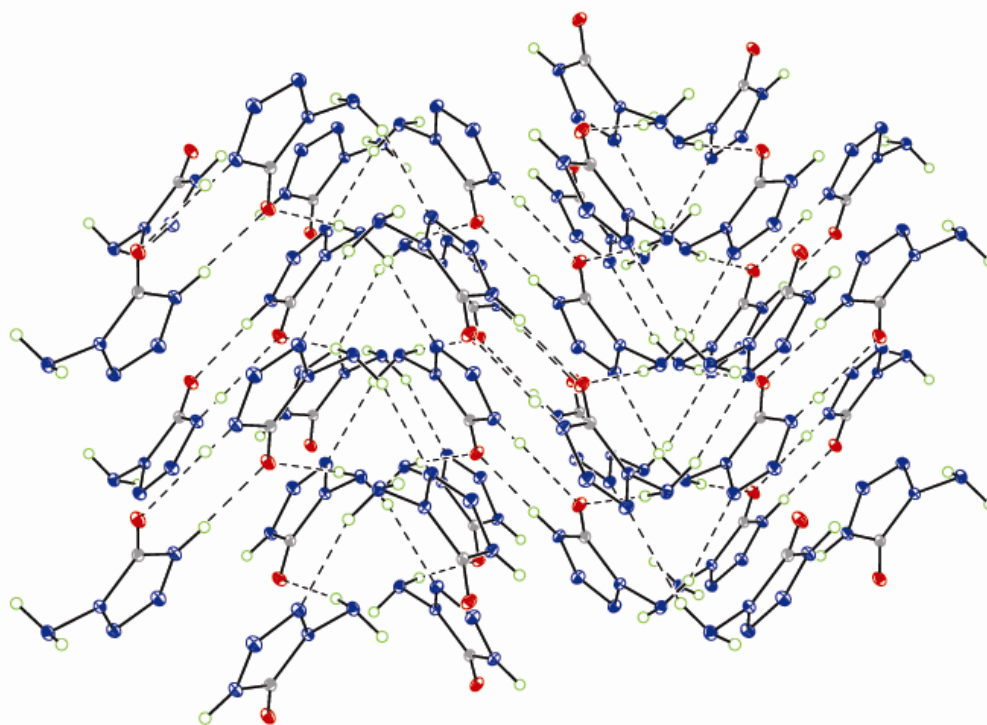


Figure S27. Packing diagram of 7 viewed down the a axis

Table S2. Hydrogen bond lengths(Å) and bond angles(°) of 7

D-H...A	d(D-H)	d(H...A)	d(D...A)	D-H...A
N4-H4...O1	0.8600	1.9600	2.807(2)	167.00
N5-H5B...N2	0.8900	2.3200	3.127(2)	151.00
N5-H5C...O1	0.8900	2.1200	2.993(2)	167.00

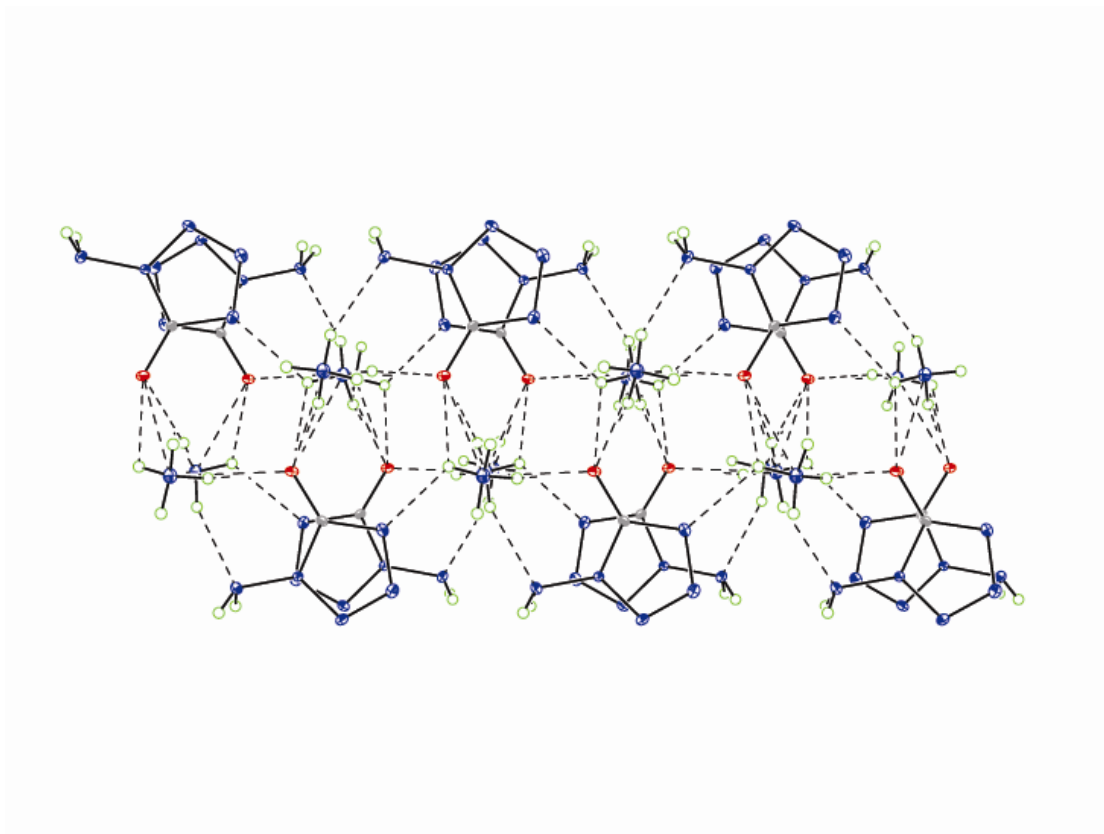


Figure S28. Packing diagram of **7a** viewed down the *c* axis

Table S3. Hydrogen bond lengths(Å) and bond angles(°) of **7a**

D-H...A	d(D-H)	d(H...A)	d(D...A)	D-H...A
N5-H5A...N2	0.8900	2.4300	3.1977	145.00
N5-H5B...N2	0.8900	2.5300	3.3468	153.00
N6-H6A...O1	0.9000	1.9600	2.8451	169.00
N6-H6B...O1	0.9000	1.9400	2.8327	169.00
N6-H6C...N5	0.9000	2.3000	3.1552	159.00
N6-H6D...N4	0.9000	2.3500	3.0157	131.00
N6-H6D...O1	0.9000	2.3900	2.9594	122.00

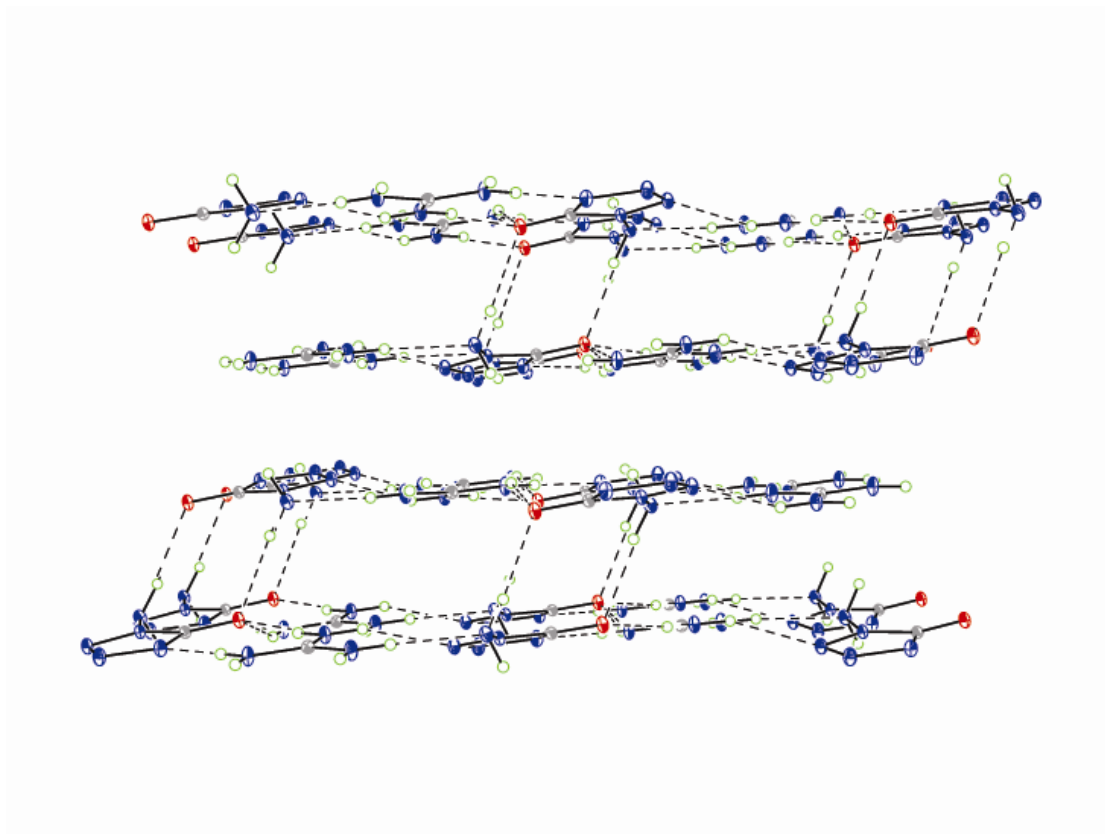


Figure S29. Packing diagram of **7b** viewed down the b axis

Table S4. Hydrogen bond lengths(Å) and bond angles(°) of **7b**

D-H...A	d(D-H)	d(H...A)	d(D...A)	D-H...A
N5-H5A...O1	0.8900	2.0500	2.9253	169.00
N5-H5B...N6	0.8900	2.6200	3.4232	150.00
N6-H6A...N5	0.8600	2.0400	2.8986	179.00
N6-H6B...O1	0.8600	2.0700	2.8840	158.00
N7-H7A...O1	0.8600	2.0400	2.9014	174.00
N7-H7B...N2	0.8600	2.3000	3.1182	159.00
N8-H8A...N5	0.8600	2.2600	3.1174	171.00

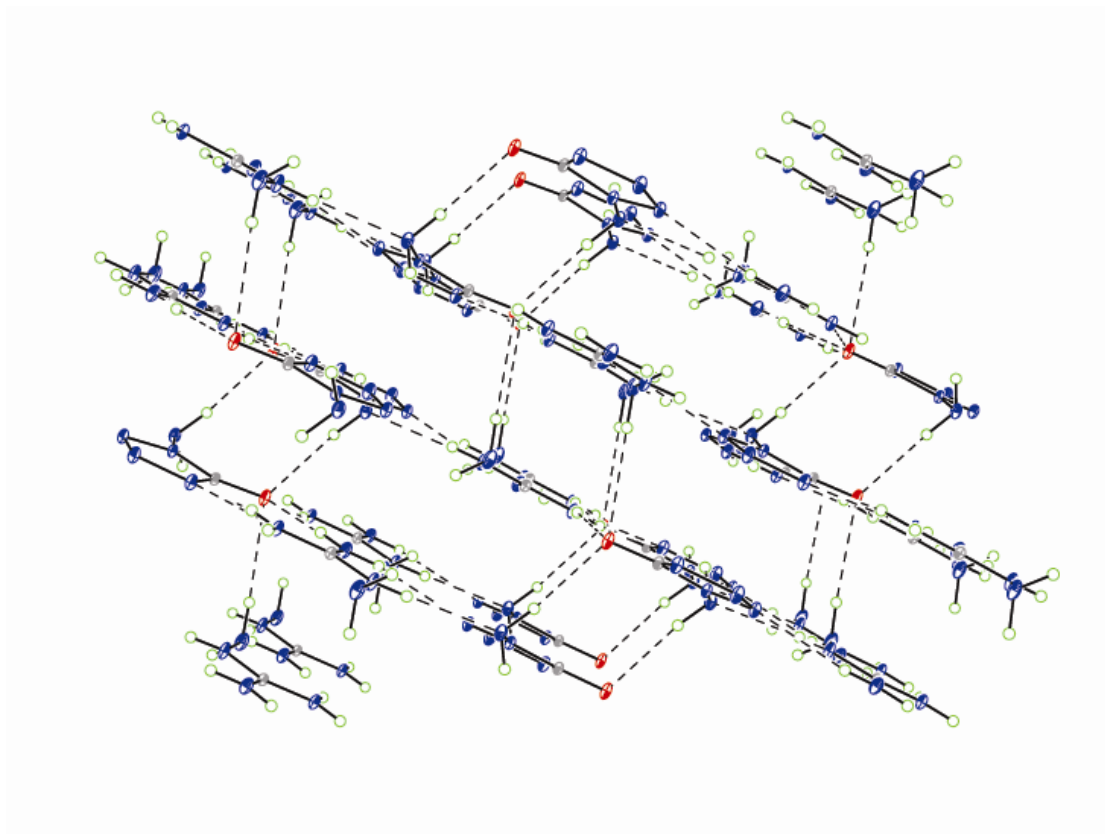


Figure S30. Packing diagram of **7c** viewed down the *b* axis

Table S5. Hydrogen bond lengths(Å) and bond angles(°) of **7c**

D-H...A	d(D-H)	d(H...A)	d(D...A)	D-H...A
N1-H1A...N6	0.8600	2.3600	2.6846	103.00
N1-H1A...O1	0.8600	2.1900	3.0237	162.00
N1-H1B...N5	0.8600	2.0800	2.9332	171.00
N6-H2...O1	0.9000	2.4600	3.3130	158.00
N4-H4...O1	0.9100	2.1100	2.9876	161.00
N7-H7A...O1	0.8600	2.0500	2.9085	175.00
N7-H7B...N3	0.8600	2.4100	3.2541	169.00
N9-H10...N4	0.8300	2.1600	2.9810	168.00

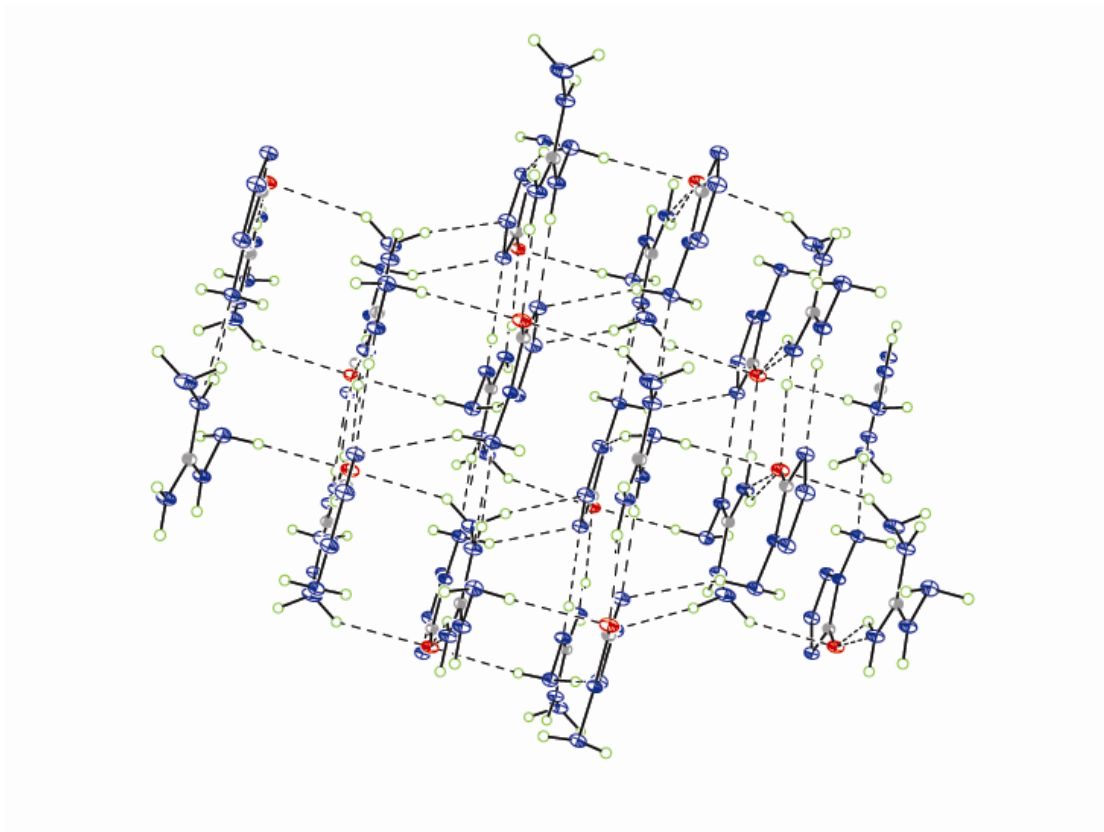


Figure S31. Packing diagram of **7d** viewed down the *b* axis

Table S6. Hydrogen bond lengths(Å) and bond angles(°) of **7d**

D-H...A	d(D-H)	d(H...A)	d(D...A)	D-H...A
N5-H5C...N4	0.8900	2.3300	3.1126	146.00
N6-H6A...N7	0.8600	2.3700	2.6982	103.00
N6-H6A...O1	0.8600	2.2100	2.9103	139.00
N6-H6B...O1	0.8600	2.0300	2.8744	169.00
N7-H7B...O1	0.8900	2.5700	3.3018	140.00
N7-H7C...N3	0.8900	2.4100	3.2610	161.00
N8-H8...N10	0.8600	2.3100	2.6536	104.00
N8-H8...N5	0.8600	2.1900	2.9927	155.00
N9-H9...N4	0.8600	2.0800	2.9305	173.00
N10-H10B...N2	0.8900	2.4100	3.2046	148.00
N10-H10C...O1	0.8900	2.3200	3.1064	148.00

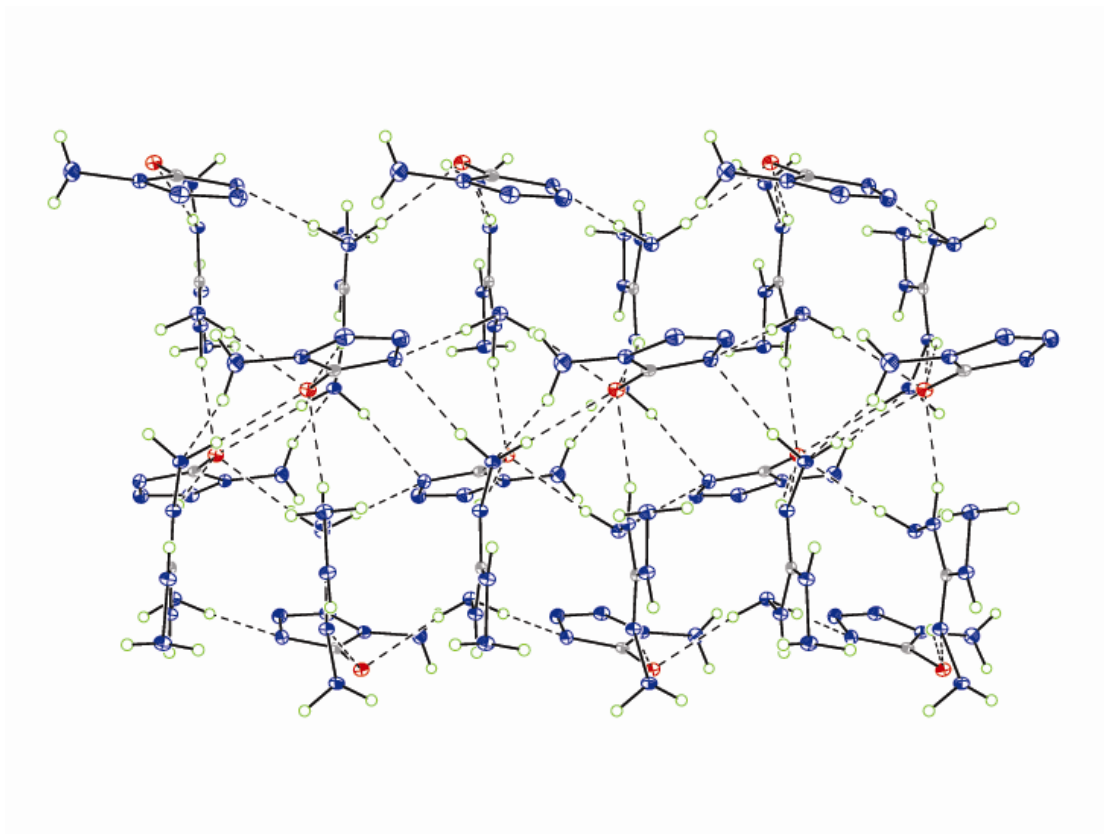


Figure S32. Packing diagram of **7e** viewed down the *c* axis

Table S7. Hydrogen bond lengths(Å) and bond angles(°) of **7e**

D-H...A	d(D-H)	d(H...A)	d(D...A)	D-H...A
N2-H2...N7	0.8600	2.5200	3.2887	148.00
N9-H4...N6	0.8400	2.3200	2.6480	104.00
N9-H4...O1	0.8400	2.1000	2.8510	149.00
N6-H5...O1	0.9100	2.2900	3.2017	174.00
N6-H6...N4	0.9300	2.2400	3.1512	164.00
N10-H7...N11	0.8800	2.3500	2.6725	102.00
N10-H7...O1	0.8800	2.1600	3.0043	159.00
N7-H8...N4	0.9200	2.1800	3.0596	162.00
N7-H9...O1	0.9000	2.2700	3.1430	163.00
N8-H10...N3	0.8500	2.3600	3.0930	146.00
N8-H10...N7	0.8500	2.2800	2.6381	105.00
N11-H11...N5	0.9100	2.4000	3.2948	166.00
N11-H12...N2	0.8900	2.3200	3.0337	136.00

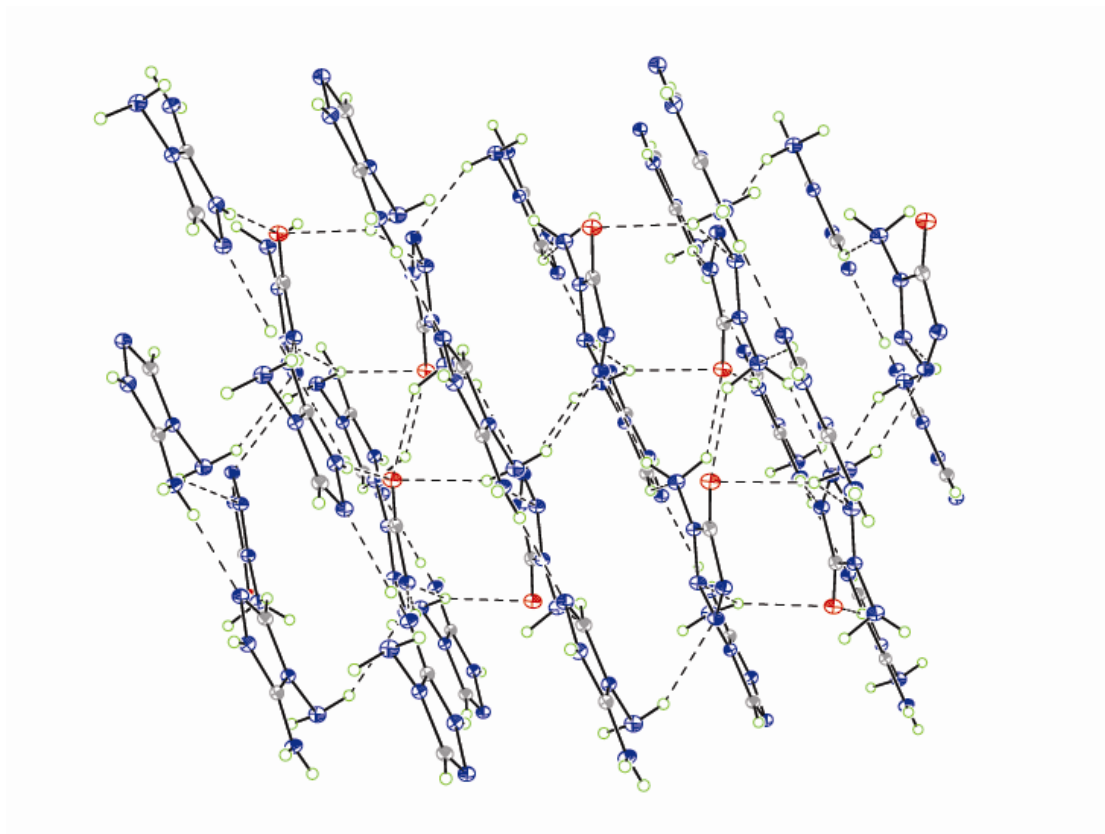


Figure S33. Packing diagram of **7f** viewed down the *c* axis

Table S8. Hydrogen bond lengths(Å) and bond angles(°) of **7f**

D-H...A	d(D-H)	d(H...A)	d(D...A)	D-H...A
N5-H5A...N3	0.8900	2.5000	3.3128	152.00
N5-H5B...O1	0.8900	2.3100	3.0268	138.00
N7-H7...O1	0.8600	1.8300	2.6624	163.00
N9-H9A...N3	0.8900	2.4000	3.2320	156.00
N9-H9B...O1	0.8900	2.3400	3.1688	155.00
N9-H9B...N2	0.8900	2.6000	3.0233	110.00
N10-H10A...N4	0.8600	2.0300	2.8652	162.00
N10-H10B...N6	0.8600	2.0700	2.8966	162.00
N10-H10B...N9	0.8600	2.5600	2.8454	100.00

TG data

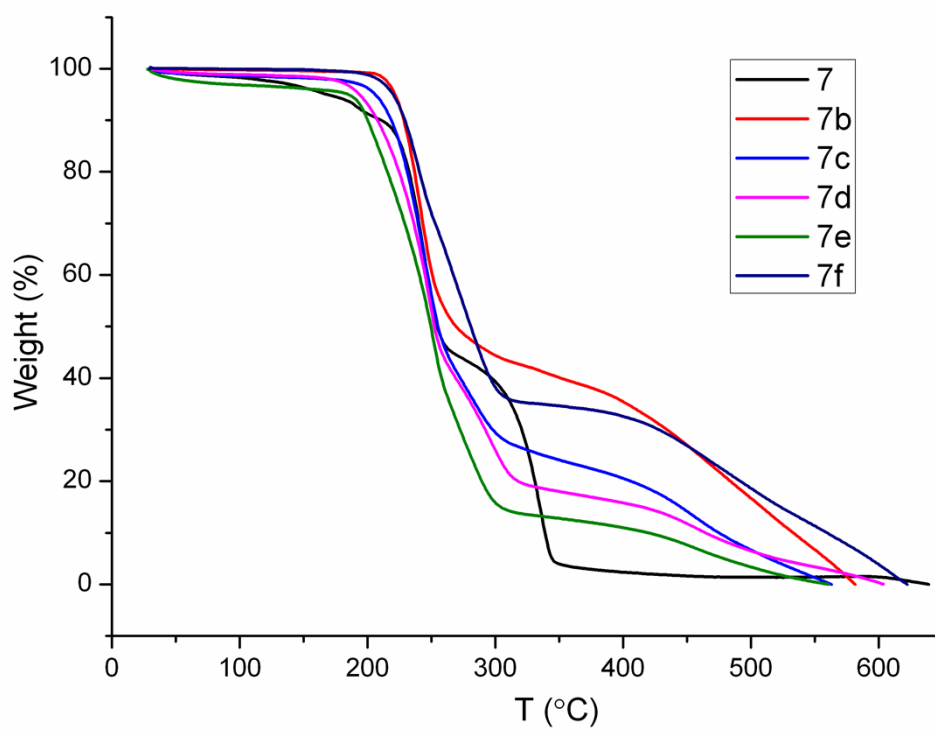


Figure S34. TG data of 7,7b~7f.