Supporting Information

For

Systematic Structural Determinants of the Effects of Tetraphosphonates on Gypsum Crystallization

By

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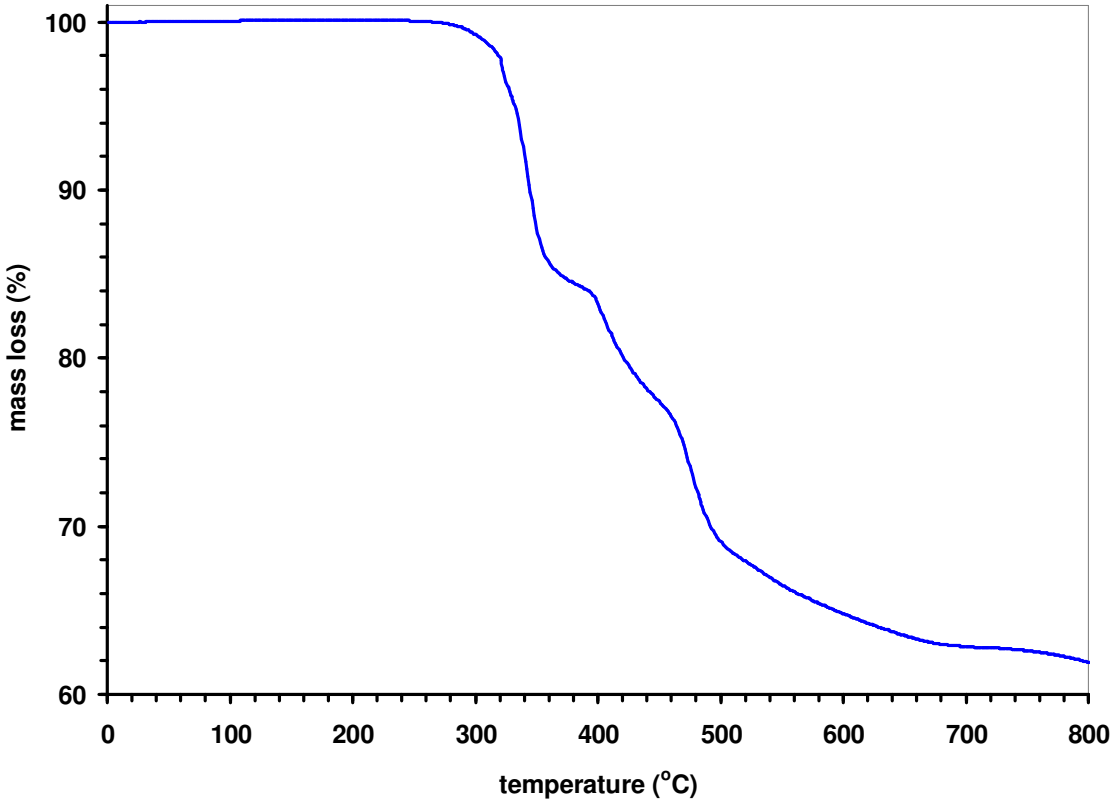
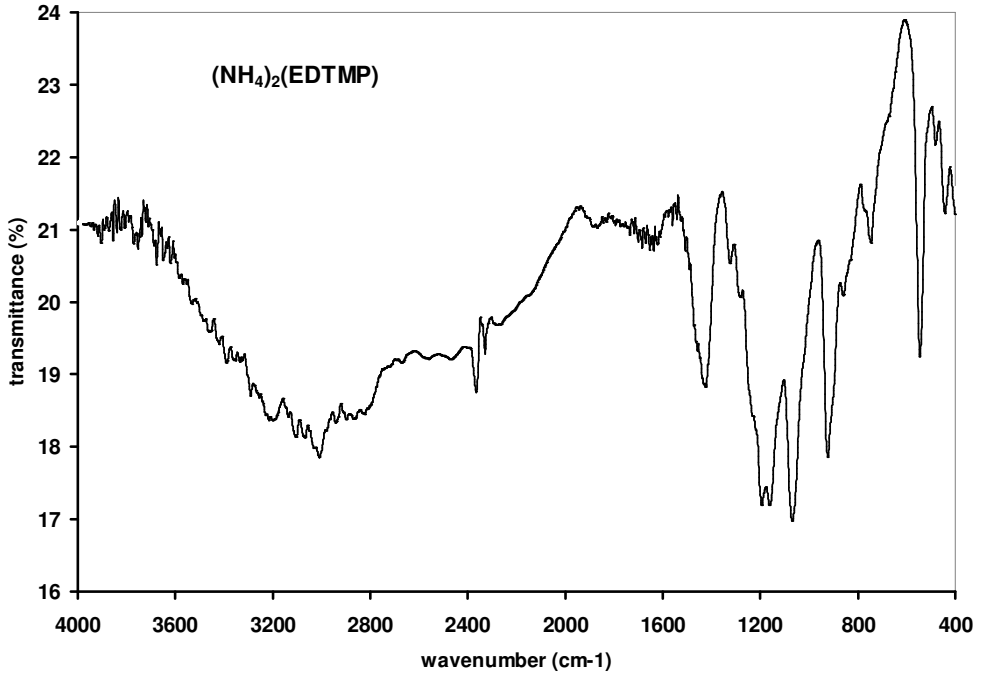
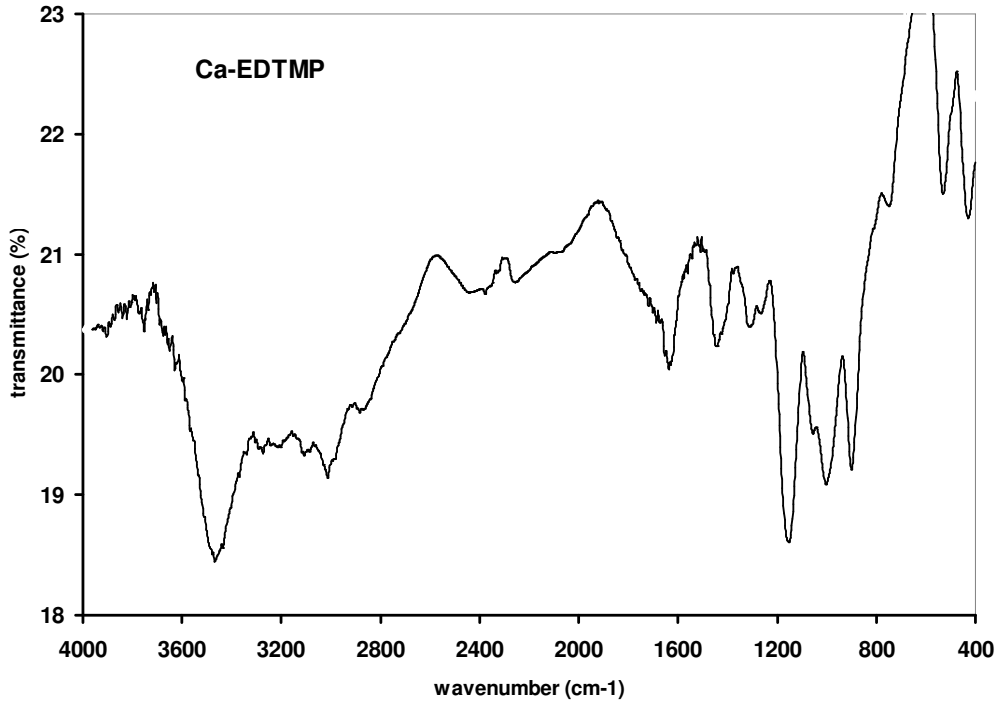
Elemental analysis for $[Ca(EDTMP)(H_2O)_2]\cdot H_2O$ (Ca-EDTMP)
Calculated for $C_6H_{24}N_2O_{15}P_4Ca$ (MW 538.24):
Calcd. C, 13.63; H, 4.90; N, 5.28
Found C, 13.92; H, 4.61; N, 5.32

TGA study of $[Ca(EDTMP)(H_2O)_2]\cdot H_2O$



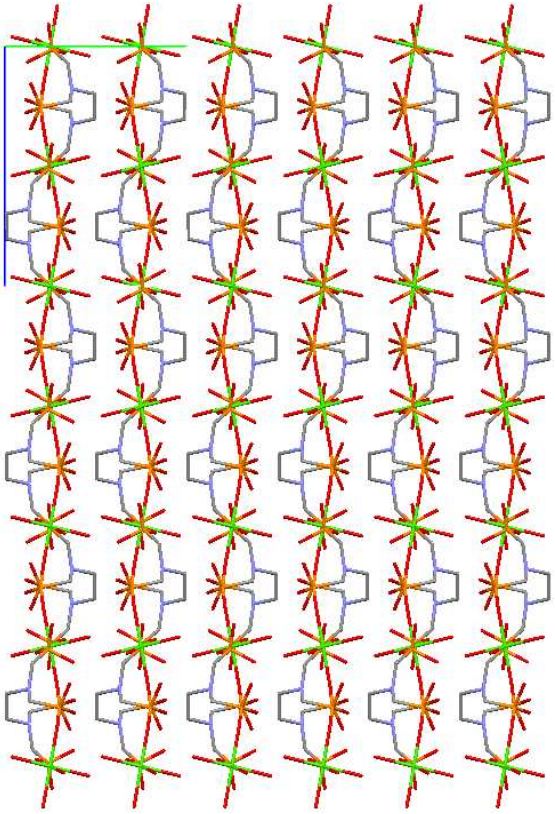
FT-IR Spectroscopy



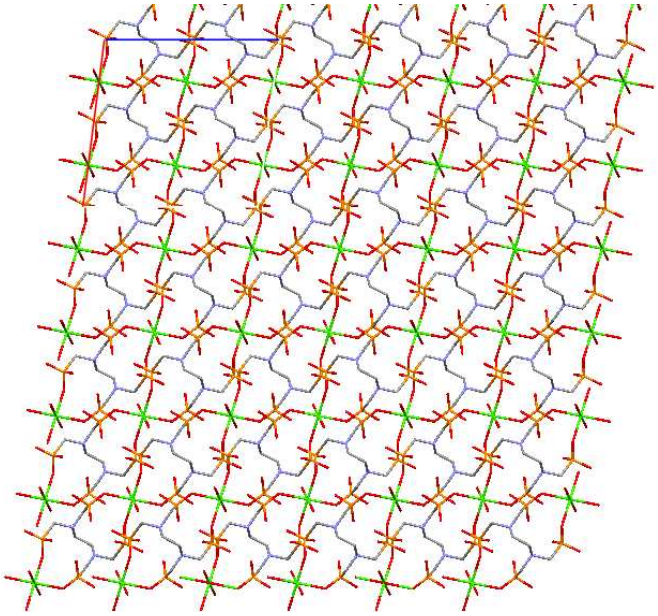


Further views of the structure of $Ca(EDTMP)(H_2O)_2]\cdot H_2O$

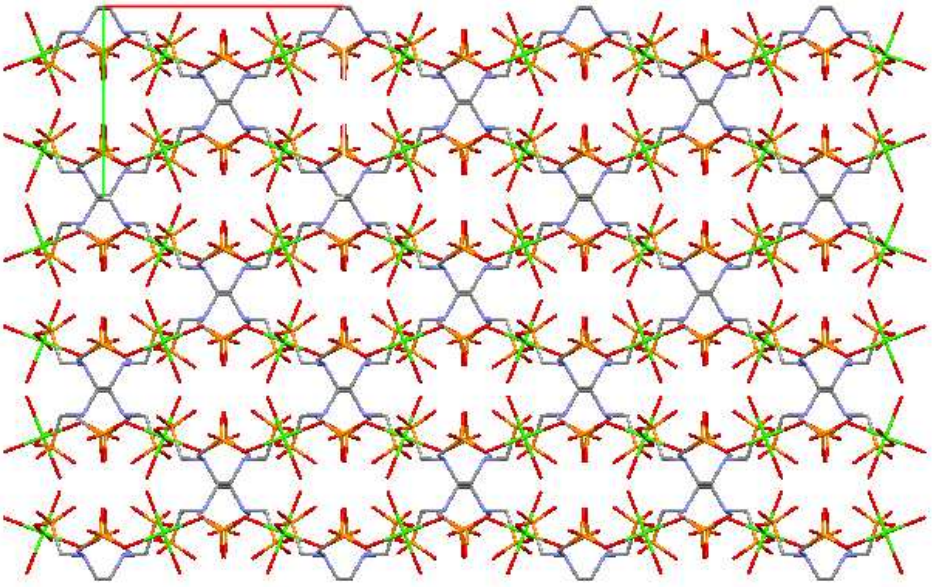
Ca-EDTMP



a-axis



b-axis



c-axis

X-ray Structural Data

Table 1. Crystal data and structure refinement for Ca-EDTMP

	Ca-EDTMP
Empirical formula	$C_6H_{24}CaN_2O_{15}P_4$
Formula weight	528.24
Temperature (K)	298(2)
Wavelength (Å)	0.71073
Crystal system	Monoclinic
Space group	C2/c
Unit cell dimensions	
a (Å)	13.206(3)
b (Å)	10.368(2)
c (Å)	13.763(3)
α (deg)	90.00
β (deg)	97.367 (18)
γ (deg)	90.00
Volume ($Å^3$)	1868.9 (7)
Z	4
Density (calc.) (Mg m^{-3})	1.877
Absorption coefficient (mm^{-1})	0.758
F(000)	1096
Crystal size (mm)	0.11 x 0.08 x 0.05
θ range for data collection (deg)	2.51 to 28.06
Index ranges	$-16 \le h \le 16$, $-7 \le k \le 13$, $-16 \le l \le 17$
Reflections collected	5926
Independent reflections	2099 [R(int) = 0.0251]
Independent reflections [$I > 2\sigma(I)$]	1766 [R(int) = 0.0248]
Max. and min. transmission	

Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	2099 / 0 / 141
Goodness-of-fit on F^2	1.077
Final *R* indices [$I > 2\sigma(I)$]	$R1 = 0.0362$, $wR2 = 0.1052$
R indices (all data)	$R1 = 0.0439$, $wR2 = 0.1094$
Largest diff. peak and hole (e Å^{-3})	0.494 and –0.384

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for **Ca-EDTMP**. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

Atom	x	y	z	U(eq)
Ca(1)	12500	2500	5000	19(1)
P(1)	9876(1)	2394(1)	5128(1)	21(1)
P(2)	7844(1)	3015(1)	7541(1)	20(1)
C(1)	9732(2)	84(2)	6976(2)	22(1)
C(2)	8919(2)	1525(3)	5698(2)	23(1)
C(3)	8235(2)	1383(2)	7282(2)	22(1)
N(1)	9167(1)	1320(2)	6772(1)	18(1)
O(1)	9960(1)	3743(2)	5651(1)	29(1)
O(2)	10889(1)	1725(2)	5275(1)	24(1)
O(3)	9389(1)	2596(2)	4087(1)	32(1)
O(4)	7257(1)	3565(2)	6563(1)	26(1)
O(5)	8787(1)	3835(2)	7769(1)	31(1)
O(7)	13177(2)	397(2)	5393(2)	42(1)
O(6)	7158(1)	2892(2)	8321(1)	28(1)
O(8)	10000	3951(4)	2500	63(1)

Table 3. Bond lengths [A] and angles [deg] for **Ca-EDTMP.**

Ca(1)-O(6)#1	2.3325(18)
Ca(1)-O(6)#2	2.3325(18)
Ca(1)-O(2)	2.3507(17)
Ca(1)-O(2)#3	2.3507(17)
Ca(1)-O(7)#3	2.392(2)
Ca(1)-O(7)	2.392(2)
Ca(1)-P(1)	3.4934(10)
Ca(1)-P(1)#3	3.4934(11)
Ca(1)-P(2)#2	3.5128(10)
Ca(1)-P(2)#1	3.5128(10)
P(1)-O(2)	1.4967(18)
P(1)-O(3)	1.5084(18)
P(1)-O(1)	1.5704(19)
P(1)-C(2)	1.811(2)
P(2)-O(6)	1.4971(18)
P(2)-O(5)	1.5074(19)
P(2)-O(4)	1.5720(17)
P(2)-C(3)	1.818(3)
P(2)-Ca(1)#2	3.5128(10)
C(1)-N(1)	1.491(3)
C(1)-C(1)#2	1.523(4)
C(1)-H(1A)	0.9700
C(1)-H(1B)	0.9700
C(2)-N(1)	1.487(3)
C(2)-H(2A)	0.9700
C(2)-H(2B)	0.9700
C(3)-N(1)	1.495(3)
C(3)-H(3A)	0.9700
C(3)-H(3B)	0.9700
N(1)-H(1E)	1.04(4)
O(1)-H(1D)	0.8200
O(4)-H(4A)	0.8200
O(7)-H(7A)	0.8200
O(7)-H(7B)	0.79(4)
O(6)-Ca(1)#2	2.3325(18)
O(8)-H(8A)	0.99(4)

O(6)#1-Ca(1)-O(6)#2	180.0
O(6)#1-Ca(1)-O(2)	92.04(6)
O(6)#2-Ca(1)-O(2)	87.96(6)
O(6)#1-Ca(1)-O(2)#3	87.96(6)
O(6)#2-Ca(1)-O(2)#3	92.04(6)
O(2)-Ca(1)-O(2)#3	180.0
O(6)#1-Ca(1)-O(7)#3	85.13(8)
O(6)#2-Ca(1)-O(7)#3	94.87(8)
O(2)-Ca(1)-O(7)#3	91.63(7)
O(2)#3-Ca(1)-O(7)#3	88.37(7)
O(6)#1-Ca(1)-O(7)	94.87(8)
O(6)#2-Ca(1)-O(7)	85.13(8)
O(2)-Ca(1)-O(7)	88.37(7)
O(2)#3-Ca(1)-O(7)	91.63(7)
O(7)#3-Ca(1)-O(7)	180.00(11)

O(6)#1-Ca(1)-P(1)	88.68(5)
O(6)#2-Ca(1)-P(1)	91.32(5)
O(2)-Ca(1)-P(1)	19.42(4)
O(2)#3-Ca(1)-P(1)	160.58(4)
O(7)#3-Ca(1)-P(1)	72.29(6)
O(7)-Ca(1)-P(1)	107.71(6)
O(6)#1-Ca(1)-P(1)#3	91.32(5)
O(6)#2-Ca(1)-P(1)#3	88.68(5)
O(2)-Ca(1)-P(1)#3	160.58(4)
O(2)#3-Ca(1)-P(1)#3	19.42(4)
O(7)#3-Ca(1)-P(1)#3	107.71(6)
O(7)-Ca(1)-P(1)#3	72.29(6)
P(1)-Ca(1)-P(1)#3	180.0
O(6)#1-Ca(1)-P(2)#2	161.48(5)
O(6)#2-Ca(1)-P(2)#2	18.52(5)
O(2)-Ca(1)-P(2)#2	70.42(5)
O(2)#3-Ca(1)-P(2)#2	109.58(5)
O(7)#3-Ca(1)-P(2)#2	89.35(6)
O(7)-Ca(1)-P(2)#2	90.65(6)
P(1)-Ca(1)-P(2)#2	72.80(2)
P(1)#3-Ca(1)-P(2)#2	107.20(2)
O(6)#1-Ca(1)-P(2)#1	18.52(5)
O(6)#2-Ca(1)-P(2)#1	161.48(5)
O(2)-Ca(1)-P(2)#1	109.58(5)
O(2)#3-Ca(1)-P(2)#1	70.42(5)
O(7)#3-Ca(1)-P(2)#1	90.65(6)
O(7)-Ca(1)-P(2)#1	89.35(6)
P(1)-Ca(1)-P(2)#1	107.20(2)
P(1)#3-Ca(1)-P(2)#1	72.80(2)
P(2)#2-Ca(1)-P(2)#1	180.000(1)
O(2)-P(1)-O(3)	117.23(11)
O(2)-P(1)-O(1)	110.01(10)
O(3)-P(1)-O(1)	107.95(11)
O(2)-P(1)-C(2)	111.80(11)
O(3)-P(1)-C(2)	104.13(11)
O(1)-P(1)-C(2)	104.87(11)
O(2)-P(1)-Ca(1)	31.48(7)
O(3)-P(1)-Ca(1)	104.63(8)
O(1)-P(1)-Ca(1)	89.05(7)
C(2)-P(1)-Ca(1)	142.13(8)
O(6)-P(2)-O(5)	117.35(12)
O(6)-P(2)-O(4)	111.56(10)
O(5)-P(2)-O(4)	106.07(10)
O(6)-P(2)-C(3)	105.94(11)
O(5)-P(2)-C(3)	108.56(11)
O(4)-P(2)-C(3)	106.91(11)
O(6)-P(2)-Ca(1)#2	29.66(7)
O(5)-P(2)-Ca(1)#2	95.23(8)
O(4)-P(2)-Ca(1)#2	140.32(7)
C(3)-P(2)-Ca(1)#2	97.05(8)
N(1)-C(1)-C(1)#2	109.83(14)
N(1)-C(1)-H(1A)	109.7
C(1)#2-C(1)-H(1A)	109.7
N(1)-C(1)-H(1B)	109.7
C(1)#2-C(1)-H(1B)	109.7
H(1A)-C(1)-H(1B)	108.2
N(1)-C(2)-P(1)	115.09(16)

N(1)-C(2)-H(2A)	108.5
P(1)-C(2)-H(2A)	108.5
N(1)-C(2)-H(2B)	108.5
P(1)-C(2)-H(2B)	108.5
H(2A)-C(2)-H(2B)	107.5
N(1)-C(3)-P(2)	113.82(16)
N(1)-C(3)-H(3A)	108.8
P(2)-C(3)-H(3A)	108.8
N(1)-C(3)-H(3B)	108.8
P(2)-C(3)-H(3B)	108.8
H(3A)-C(3)-H(3B)	107.7
C(2)-N(1)-C(1)	110.57(18)
C(2)-N(1)-C(3)	111.76(17)
C(1)-N(1)-C(3)	111.72(18)
C(2)-N(1)-H(1E)	114(3)
C(1)-N(1)-H(1E)	107(2)
C(3)-N(1)-H(1E)	101(3)
P(1)-O(1)-H(1D)	109.5
P(1)-O(2)-Ca(1)	129.10(10)
P(2)-O(4)-H(4A)	109.5
Ca(1)-O(7)-H(7A)	109.5
Ca(1)-O(7)-H(7B)	127(3)
H(7A)-O(7)-H(7B)	122.9
P(2)-O(6)-Ca(1)#2	131.82(11)

Symmetry transformations used to generate equivalent atoms:
#1 x+1/2,-y+1/2,z-1/2 #2 -x+2,y,-z+3/2 #3 -x+5/2,-y+1/2,-z+1

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for **Ca-EDTMP.** The anisotropic displacement factor exponent takes the form: $-2\ pi^2\ [\ h^2\ a^{*2}\ U11 + ... + 2\ h\ k\ a^*\ b^*\ U12\ ]$

Atom	U11	U22	U33	U23	U13	U12
Ca(1)	17(1)	24(1)	15(1)	-1(1)	5(1)	-2(1)
P(1)	19(1)	27(1)	16(1)	1(1)	3(1)	-2(1)
P(2)	20(1)	24(1)	16(1)	1(1)	3(1)	2(1)
C(1)	22(1)	20(1)	23(1)	-3(1)	4(1)	1(1)
C(2)	18(1)	32(1)	17(1)	1(1)	1(1)	-3(1)
C(3)	23(1)	23(1)	21(1)	3(1)	8(1)	1(1)
N(1)	19(1)	20(1)	17(1)	1(1)	4(1)	0(1)
O(1)	32(1)	25(1)	29(1)	0(1)	-4(1)	2(1)
O(2)	19(1)	29(1)	26(1)	1(1)	7(1)	-1(1)
O(3)	30(1)	47(1)	19(1)	8(1)	-1(1)	-8(1)
O(4)	27(1)	30(1)	19(1)	6(1)	0(1)	1(1)
O(5)	31(1)	29(1)	31(1)	3(1)	-2(1)	-5(1)
O(7)	36(1)	32(1)	61(2)	13(1)	18(1)	7(1)
O(6)	28(1)	40(1)	18(1)	5(1)	7(1)	10(1)
O(8)	94(3)	52(2)	47(2)	0	30(2)	0

Table 5. Hydrogen coordinates ($x10^4$) and isotropic displacement parameters ($A^2 \times 10^3$) for Ca-EDTMP.

Atom	x	y	z	U(eq)
H(1A)	10231	-18	6523	26
H(1B)	9258	-634	6884	26
H(2A)	8280	1994	5578	27
H(2B)	8815	690	5383	27
H(3A)	8365	911	7894	26
H(3B)	7677	957	6879	26
H(1D)	10373	3700	6148	44
H(4A)	6739	3138	6406	38
H(7A)	12805	17	5732	63
H(1E)	9620(30)	2040(40)	7130(30)	86(16)
H(7B)	13700(30)	110(40)	5270(30)	57(12)
H(8A)	9890(30)	3320(40)	3010(30)	57(11)