Formation of diphosphates. A NMR study on the mechanism and stereochemistry of diphosphate formation from chiral dioxaphosphorinanes

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S U P P L E M E N T A R Y M A T E R I A L
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B E L·O N G I N G T O T H E P A P E R

The Formation of Pyrophosphates; An NMR Study on the Mechanism and Stereochemistry of Pyrophosphate Formation from Chiral Dioxaphosphorinanes

b y

Ron Hulst, Johanna M. Visser, N. Koen de Vries, Robert W.J. Zijlstra, Huub Kooijman, Wilberth J.J. Smeets, Anthony L. Spek and Ben L. Feringa

COMPOUND 12

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Table S1 - Crystal Data and Details of the Structure Determination for: S691A

Crystal Data			
Empirical Formula			C20 H25 O4 P
Formula Weight			360.37
Crystal System			Orthorhombic
Space group		P212121	(No. 19)
a, b, c [Angstrom]	6.4799(10)	17.165(2)	17.189(2)
V [Ang**3]			1911.9(4)
Z			4
D(calc) [g/cm**3]			1.252
F(000)			768
Mu(MoKa) [/mm]			0.2
Crystal Size [mm]			0.18 x 0.45 x 1.12
Data Collection			
Temperature (K)			295
Radiation [Angstrom]			MoKa (Ni filter) 0.71073
Scan,(Type & Range) [Deg]			Omega / 2 Theta, 0.66 + 0.35 Tan(Theta)
Hor. and vert. aperture [mm]			2.30 4.00
Theta Min-Max [Deg]			1.2, 25.0
Dataset			0: 8 ; -22: 0 ; -22: 22
Tot., Uniq. Data			1966, 1958
Refinement			
Nref, Npar			1958, 246
wR2, R1, S	0.1001, 0.0511 [for 1061 F > 4 sigma(F)], 0.923		
w	[sigma**2(F)+(0.0416P)**2]**-1, P=(Max(Fo**2,0)+2Fc**2)/3		
Max. and Av. Shift/Error			0.000, 0.000
Min. and Max. resd. dens. [e/Ang^3]			-0.22, 0.19

able S2 - Final Coordinates and Equivalent Isotropic Displacement Parameters of the non-Hydrogen atoms
for: S691A

tom	x	y	z	U(eq) [Ang^2]
1	0.8230(2)	0.11286(8)	0.48444(9)	0.0458(5)
01	0.7471(7)	0.1396(2)	0.4101(2)	0.0683(14)
02	0.7368(6)	0.1547(2)	0.5582(2)	0.0503(14)
03	1.0648(5)	0.1169(2)	0.4897(2)	0.0553(12)
04	0.7667(5)	0.0258(2)	0.5023(2)	0.0420(12)
C1	1.1668(9)	0.0766(3)	0.5529(3)	0.057(2)
C2	1.1047(8)	-0.0098(3)	0.5565(3)	0.044(2)
C3	1.1708(9)	-0.0513(3)	0.4824(3)	0.061(2)
C4	1.2102(8)	-0.0436(3)	0.6278(3)	0.067(2)
C5	0.8677(8)	-0.0116(3)	0.5697(3)	0.0430(19)
C6	0.7746(8)	-0.0918(3)	0.5778(3)	0.0420(19)
C7	0.7020(8)	-0.1345(3)	0.5156(4)	0.0520(17)
C8	0.6102(9)	-0.2061(3)	0.5272(4)	0.067(3)
C9	0.5851(11)	-0.2346(4)	0.6000(5)	0.082(3)
C10	0.6566(10)	-0.1935(4)	0.6634(4)	0.073(3)
C11	0.7474(9)	-0.1207(3)	0.6526(3)	0.060(2)
C12	0.8088(10)	0.2312(3)	0.5834(3)	0.056(2)
C13	0.7085(10)	0.2459(3)	0.6624(3)	0.058(2)
C14	0.7787(11)	0.3267(3)	0.6909(3)	0.081(3)
C15	0.7516(11)	0.1802(3)	0.7176(3)	0.061(2)
C16	0.9432(13)	0.1722(4)	0.7527(4)	0.078(3)
C17	0.9846(14)	0.1087(5)	0.7992(4)	0.099(4)
C18	0.8395(18)	0.0519(5)	0.8119(4)	0.101(4)
C19	0.6483(14)	0.0601(5)	0.7784(4)	0.095(4)
C20	0.6058(11)	0.1241(4)	0.7314(3)	0.071(3)

U(eq) = 1/3 of the trace of the orthogonalized U Tensor

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Table S3 - Hydrogen Atom Positions and Isotropic Displacement Parameters
for: S691A

Atom	x	y	z	U(iso) [Ang^2]
H11	1.1328(14)	0.1005(9)	0.5997(18)	0.0770
H12	1.309(5)	0.0803(3)	0.5465(3)	0.0770
H31	1.096(4)	-0.0278(14)	0.4365(13)	0.0910
H32	1.136(4)	-0.1084(16)	0.4865(8)	0.0910
H33	1.324(4)	-0.0452(15)	0.4752(10)	0.0910
H41	1.152(4)	-0.0181(16)	0.6765(14)	0.1010
H42	1.365(5)	-0.0334(17)	0.6246(10)	0.1010
H43	1.184(4)	-0.1021(17)	0.6300(11)	0.1010
H51	0.8376(16)	0.0165(13)	0.613(2)	0.0520
H71	0.7144(10)	-0.1156(10)	0.467(2)	0.0620
H81	0.565(3)	-0.2355(18)	0.484(3)	0.0800
H91	0.513(4)	-0.287(3)	0.6080(7)	0.0980
H101	0.6444(12)	-0.2141(12)	0.713(3)	0.0870
H111	0.792(2)	-0.0903(15)	0.697(2)	0.0720
H121	0.7648(16)	0.2738(14)	0.5437(14)	0.0750
H122	0.969(5)	0.2317(3)	0.5883(3)	0.0750
H131	0.543(8)	0.2483(3)	0.6536(5)	0.0700
H141	0.753(6)	0.3645(12)	0.6513(16)	0.1210
H142	0.923(5)	0.3255(7)	0.702(2)	0.1210
H143	0.704(5)	0.3404(12)	0.7368(19)	0.1210
H161	1.062(7)	0.217(2)	0.7434(6)	0.0930
H171	1.117(10)	0.1042(6)	0.8230(17)	0.1190
H181	0.870(3)	0.009(3)	0.843(2)	0.1210
H191	0.570(8)	0.031(3)	0.7852(8)	0.1130
H201	0.483(8)	0.1287(5)	0.7102(14)	0.0850

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The Temperature Factor has the Form of Exp(-T) Where
T = 8*(Pi**2)*U*(Sin(Theta)/Lambda)**2 for Isotropic Atoms

e S4 - (An)isotropic Displacement Parameters
for: S691A

U(1,1) or U	U(2,2)	U(3,3)	U(2,3)	U(1,3)	U(1,2)
0.0516(10)	0.0398(8)	0.0461(9)	-0.0016(8)	-0.0050(8)	0.0034(8)
0.094(3)	0.065(2)	0.046(2)	0.009(2)	-0.017(2)	0.009(3)
0.054(3)	0.039(2)	0.058(2)	-0.008(2)	0.000(2)	0.005(2)
0.050(2)	0.055(2)	0.061(2)	0.008(2)	0.005(2)	-0.003(2)
0.041(2)	0.039(2)	0.046(2)	-0.003(2)	-0.013(2)	0.004(2)
0.038(3)	0.067(4)	0.066(4)	0.002(3)	-0.004(3)	-0.007(3)
0.035(4)	0.052(4)	0.046(3)	-0.003(3)	0.000(3)	0.006(3)
0.050(4)	0.063(3)	0.069(4)	-0.002(3)	0.009(4)	0.010(3)
0.044(4)	0.080(4)	0.078(4)	0.018(3)	-0.021(4)	0.014(4)
0.044(4)	0.045(3)	0.040(3)	0.002(3)	-0.005(3)	0.005(3)
0.028(3)	0.039(3)	0.059(4)	0.010(3)	0.001(3)	0.012(3)
0.045(3)	0.047(3)	0.064(3)	-0.003(3)	-0.001(4)	0.004(3)
0.054(4)	0.047(4)	0.099(5)	0.003(4)	-0.003(4)	0.000(3)
0.071(5)	0.049(4)	0.125(6)	0.019(5)	0.017(5)	-0.003(4)
0.056(5)	0.071(5)	0.092(5)	0.037(4)	0.021(5)	0.013(4)
0.049(4)	0.067(4)	0.063(4)	0.011(4)	0.002(3)	0.002(4)
0.076(5)	0.040(3)	0.051(3)	-0.005(3)	0.003(4)	0.006(4)
0.060(4)	0.054(3)	0.061(4)	-0.013(3)	-0.004(4)	0.003(3)
0.108(6)	0.064(4)	0.070(4)	-0.026(3)	0.001(4)	-0.001(4)
0.070(5)	0.071(4)	0.042(3)	-0.016(3)	0.009(4)	-0.002(4)
0.083(6)	0.093(5)	0.057(4)	0.004(4)	-0.015(5)	-0.009(5)
0.099(7)	0.131(7)	0.067(5)	0.016(5)	-0.015(5)	0.007(7)
0.158(9)	0.103(6)	0.042(4)	0.009(4)	0.008(6)	0.003(7)
0.127(9)	0.085(6)	0.072(6)	-0.008(5)	0.035(6)	-0.036(6)
0.067(5)	0.078(5)	0.067(4)	-0.020(4)	0.009(4)	-0.011(5)

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The Temperature Factor has the Form of Exp(-T) Where
T = 8*(Pi**2)*U*(Sin(Theta)/Lambda)**2 for Isotropic Atoms
T = 2*(Pi**2)*Sumij(h(i)*h(j)*U(i,j)*Astar(i)*Astar(j)), for
nisotropic Atoms. Astar(i) are Reciprocal Axial Lengths and
h(i) are the Reflection Indices.

Table S5 - Bond Distances (Angstrom)
for: S691A

P1	-O1	1.444(4)	C19	-C20	1.391(10)
P1	-O2	1.561(4)	C1	-H11	0.93(3)
P1	-O3	1.571(4)	C1	-H12	0.93(3)
P1	-O4	1.569(4)	C3	-H31	1.01(2)
O2	-C12	1.459(6)	C3	-H32	1.01(3)
O3	-C1	1.448(6)	C3	-H33	1.01(3)
O4	-C5	1.477(6)	C4	-H41	1.02(3)
C1	-C2	1.538(7)	C4	-H42	1.02(3)
C2	-C3	1.521(7)	C4	-H43	1.02(3)
C2	-C4	1.519(7)	C5	-H51	0.91(3)
C2	-C5	1.553(7)	C7	-H71	0.90(3)
C5	-C6	1.509(7)	C8	-H81	0.94(4)
C6	-C7	1.379(8)	C9	-H91	1.02(5)
C6	-C11	1.389(7)	C10	-H101	0.93(5)
C7	-C8	1.380(7)	C11	-H111	0.97(3)
C8	-C9	1.353(11)	C12	-H121	1.04(2)
C9	-C10	1.378(11)	C12	-H122	1.04(3)
C10	-C11	1.394(9)	C13	-H131	1.08(5)
C12	-C13	1.526(8)	C14	-H141	0.96(3)
C13	-C14	1.540(8)	C14	-H142	0.95(3)
C13	-C15	1.500(7)	C14	-H143	0.96(3)
C15	-C16	1.387(11)	C16	-H161	1.10(4)
C15	-C20	1.370(9)	C17	-H171	0.95(6)
C16	-C17	1.378(11)	C18	-H181	0.93(5)
C17	-C18	1.372(13)	C19	-H191	0.72(5)
C18	-C19	1.373(14)	C20	-H201	0.88(5)

Sub 3

e S6 - Bond Angles (Degrees)
for: S691A

-P1	-O2	116.8(2)	C12	-C13	-C14	107.8(5)
-P1	-O3	112.1(2)	C12	-C13	-C15	111.0(5)
-P1	-O4	113.4(2)	C14	-C13	-C15	114.9(5)
-P1	-O3	106.9(2)	C13	-C15	-C16	121.1(6)
-P1	-O4	101.3(2)	C13	-C15	-C20	120.6(6)
-P1	-O4	105.23(19)	C16	-C15	-C20	118.2(5)
-O2	-C12	122.7(3)	C15	-C16	-C17	120.3(7)
-O3	-C1	118.5(3)	C16	-C17	-C18	121.4(8)
-O4	-C5	117.7(3)	C17	-C18	-C19	118.6(8)
-C1	-C2	111.8(4)	C18	-C19	-C20	120.2(8)
-C2	-C3	110.1(4)	C15	-C20	-C19	121.3(7)
-C2	-C4	106.4(4)	O3	-C1	-H11	109.3(13)
-C2	-C5	106.5(4)	O3	-C1	-H12	109.3(6)
-C2	-C4	111.7(4)	C2	-C1	-H11	109.2(12)
-C2	-C5	113.1(4)	C2	-C1	-H12	109.3(6)
-C2	-C5	108.6(4)	H11	-C1	-H12	107.9(9)
-C5	-C2	108.4(4)	C2	-C3	-H31	109.4(14)
-C5	-C6	107.0(4)	C2	-C3	-H32	109.5(10)
-C5	-C6	115.3(4)	C2	-C3	-H33	109.4(12)
-C6	-C7	123.3(5)	H31	-C3	-H32	109.6(18)
-C6	-C11	117.5(5)	H31	-C3	-H33	109.6(19)
-C6	-C11	119.0(5)	H32	-C3	-H33	109(2)
-C7	-C8	120.6(6)	C2	-C4	-H41	109.4(15)
-C8	-C9	120.5(6)	C2	-C4	-H42	109.5(12)
-C9	-C10	120.4(6)	C2	-C4	-H43	109.4(13)
-C10	-C11	119.7(6)	H41	-C4	-H42	109.6(19)
-C11	-C10	119.8(5)	H41	-C4	-H43	109(2)
-C12	-C13	106.1(4)	H42	-C4	-H43	110(2)

Table S6 - Bond Angles (Degrees) (continued)
for: S691A

O4	-C5	-H51	108.5(16)	C14	-C13	-H131	107.6(6)
C2	-C5	-H51	108.8(9)	C15	-C13	-H131	107.5(7)
C6	-C5	-H51	108.8(15)	C13	-C14	-H141	109.5(17)
C6	-C7	-H71	119.9(13)	C13	-C14	-H142	109.5(10)
C8	-C7	-H71	119.6(14)	C13	-C14	-H143	109.6(15)
C7	-C8	-H81	120(2)	H141	-C14	-H142	109(3)
C9	-C8	-H81	120(2)	H141	-C14	-H143	109(2)
C8	-C9	-H91	119.8(10)	H142	-C14	-H143	110(3)
C10	-C9	-H91	119.8(11)	C15	-C16	-H161	119.6(19)
C9	-C10	-H101	120.2(17)	C17	-C16	-H161	120.1(19)
C11	-C10	-H101	120.1(17)	C16	-C17	-H171	119.2(17)
C6	-C11	-H111	119.9(18)	C18	-C17	-H171	119.4(16)
C10	-C11	-H111	120.3(18)	C17	-C18	-H181	120.5(19)
O2	-C12	-H121	110.5(13)	C19	-C18	-H181	120.9(19)
O2	-C12	-H122	110.5(5)	C18	-C19	-H191	120(4)
C13	-C12	-H121	110.5(12)	C20	-C19	-H191	120(4)
C13	-C12	-H122	110.6(6)	C15	-C20	-H201	119.3(14)
H121	-C12	-H122	108.7(9)	C19	-C20	-H201	119.4(15)
C12	-C13	-H131	107.7(7)				

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S886B : Aug 12 09:17:35 1999

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S U P P L E M E N T A R Y M A T E R I A L
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B E L O N G I N G T O T H E P A P E R

The Formation of Pyrophosphates; An NMR Study on the Mechanism and Stereochemistry of Pyrophosphate Formation from Chiral Dioxaphosphorinanes

b y

Ron Hulst, Johanna M. Visser, N. Koen de Vries, Robert W.J. Zijlstra, Huub Kooijman, Wilberth J.J. Smeets, Anthony L. Spek and Ben L. Feringa

| C O M P O U N D 13 |

C o n t e n t s
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Table S1 - Crystal Data and Details of the Structure Determination for: S886B

Crystal Data

Empirical Formula			C19 H24 N O3 P
Formula Weight			345.36
Crystal System			Trigonal
Space group		P31	(No.144)
a, b, c [Angstrom]	12.4170(12)	12.4170(12)	10.912(2)
alpha, beta, gamma [deg]	90	90	120
V [Ang**3]			1457.0(3)
Z			3
D(calc) [g/cm**3]			1.181
F(000)			552
Mu(CuKa) [/mm]			1.4
Crystal Size [mm]		0.05 x 0.05 x	0.70

Data Collection

Temperature (K)			295
Radiation [Angstrom]		CuKa (Zr filter)	1.54184
Theta Min-Max [Deg]			4.1, 75.0
Scan,(Type & Range) [Deg]	Omega / 2 Theta, 0.62 + 0.14 Tan(Theta)		
Hor. and vert. aperture [mm]		3.93	6.00
Dataset		-15: 15 ; -15: 15 ; -13: 13	
Tot., Uniq. Data, R(int)		5742, 3828,	0.058

Refinement

Npar	223
wR2, R1, S	0.1678, 0.0631 [for 2417 F > 4 sigma(F)], 0.983
w	[sigma**2(F)+(0.0879P)**2]**-1, P=(Max(Fo**2,0)+2Fc**2)/3
Max. and Av. Shift/Error	0.008, 0.001
Min. and Max. resd. dens. [e/Ang^3]	-0.18, 0.19

Table S2 - Final Coordinates and Equivalent Isotropic Displacement Parameters of the non-Hydrogen atoms
for: S886B

Atom	x	y	z	U(eq) [Ang^2]
P1	0.07639(10)	0.16672(10)	0.97802(9)	0.0641(4)
O1	0.0045(3)	0.0326(3)	1.0050(3)	0.0856(12)
O2	0.2038(3)	0.2367(3)	1.0492(2)	0.0764(12)
O3	0.0108(2)	0.2436(3)	1.0184(2)	0.0628(11)
N1	0.1094(4)	0.1962(4)	0.8361(4)	0.0746(14)
C1	0.1215(13)	0.4702(8)	0.7112(12)	0.235(7)
C2	0.0463(18)	0.5081(10)	0.6462(16)	0.295(10)
C3	-0.0626(11)	0.4231(12)	0.6059(10)	0.159(5)
C4	-0.0897(7)	0.3055(10)	0.6011(7)	0.137(4)
C5	-0.0149(6)	0.2699(7)	0.6611(7)	0.110(3)
C6	0.0918(6)	0.3548(5)	0.7181(4)	0.0792(18)
C7	0.1780(5)	0.3186(4)	0.7813(4)	0.0758(16)
C8	0.2752(6)	0.3210(7)	0.6922(6)	0.116(3)
C9	0.1911(5)	0.2505(6)	1.1802(4)	0.0884(19)
C10	0.1284(4)	0.3263(5)	1.2090(4)	0.0778(19)
C11	0.2092(5)	0.4586(5)	1.1634(6)	0.100(2)
C12	0.1148(6)	0.3239(8)	1.3481(5)	0.119(3)
C13	0.0004(4)	0.2616(4)	1.1494(4)	0.0621(12)
C14	-0.0708(4)	0.3297(4)	1.1598(4)	0.0657(16)
C15	-0.1504(5)	0.3074(6)	1.2580(5)	0.091(2)
C16	-0.2120(5)	0.3754(7)	1.2715(6)	0.106(3)
C17	-0.1958(6)	0.4616(7)	1.1889(8)	0.112(3)
C18	-0.1196(6)	0.4844(6)	1.0921(7)	0.107(3)
C19	-0.0602(5)	0.4168(5)	1.0770(6)	0.0869(19)

U(eq) = 1/3 of the trace of the orthogonalized U Tensor

Table S3 - Hydrogen Atom Positions and Isotropic Displacement Parameters
for: S886B

Atom	x	y	z	U(iso) [Ang^2]
H1	0.1936(13)	0.5300(8)	0.7497(12)	0.2820
H1A	0.079(5)	0.134(6)	0.777(6)	0.1120
H2	0.0744(18)	0.5920(10)	0.6326(16)	0.3520
H3	-0.1207(11)	0.4449(12)	0.5808(10)	0.1910
H4	-0.1588(7)	0.2473(10)	0.5575(7)	0.1640
H5	-0.0376(6)	0.1863(7)	0.6626(7)	0.1320
H7	0.2221(5)	0.3789(4)	0.8468(4)	0.0910
H8A	0.319(3)	0.4010(16)	0.653(3)	0.1740
H8B	0.2342(7)	0.258(3)	0.631(3)	0.1740
H8C	0.333(3)	0.306(5)	0.7366(9)	0.1740
H9A	0.2729(5)	0.2905(6)	1.2175(4)	0.1070
H9B	0.1428(5)	0.1686(6)	1.2166(4)	0.1070
H11A	0.2914(12)	0.4926(14)	1.197(3)	0.1500
H11B	0.174(2)	0.5083(11)	1.189(3)	0.1500
H11C	0.213(3)	0.4586(7)	1.0755(6)	0.1500
H12A	0.1957(6)	0.365(4)	1.3852(5)	0.1780
H12B	0.070(4)	0.2392(8)	1.3758(7)	0.1780
H12C	0.071(4)	0.366(4)	1.3707(6)	0.1780
H13	-0.0490(4)	0.1796(4)	1.1875(4)	0.0740
H15	-0.1630(5)	0.2468(6)	1.3154(5)	0.1100
H16	-0.2643(5)	0.3604(7)	1.3382(6)	0.1270
H17	-0.2372(6)	0.5062(7)	1.1980(8)	0.1340
H18	-0.1073(6)	0.5456(6)	1.0356(7)	0.1280
H19	-0.0110(5)	0.4311(5)	1.0079(6)	0.1040

The Temperature Factor has the Form of Exp(-T) Where
T = 8*(Pi**2)*U*(Sin(Theta)/Lambda)**2 for Isotropic Atoms

le S4 - (An)isotropic Displacement Parameters
for: S886B

n	U(1,1) or U	U(2,2)	U(3,3)	U(2,3)	U(1,3)	U(1,2)
	0.0712(7)	0.0632(7)	0.0603(5)	0.0034(5)	0.0142(5)	0.0353(6)
	0.108(2)	0.061(2)	0.082(2)	0.012(2)	0.035(2)	0.038(2)
	0.068(2)	0.100(2)	0.072(2)	0.000(2)	0.0054(15)	0.050(2)
	0.061(2)	0.070(2)	0.0578(15)	-0.0099(13)	0.0000(13)	0.0330(14)
	0.089(3)	0.062(2)	0.065(2)	0.005(2)	0.022(2)	0.032(2)
	0.343(15)	0.079(5)	0.285(13)	-0.057(6)	-0.195(12)	0.107(7)
	0.41(2)	0.102(7)	0.40(2)	-0.069(10)	-0.24(2)	0.149(11)
	0.202(10)	0.171(9)	0.175(8)	0.010(7)	0.018(7)	0.146(9)
	0.109(5)	0.196(9)	0.116(5)	0.044(5)	0.022(4)	0.084(6)
	0.108(4)	0.103(5)	0.117(4)	0.036(4)	0.010(4)	0.051(4)
	0.116(4)	0.064(3)	0.059(2)	-0.004(2)	0.007(3)	0.046(3)
	0.089(3)	0.064(3)	0.066(2)	0.009(2)	0.012(2)	0.032(2)
	0.099(4)	0.134(5)	0.121(5)	0.051(4)	0.050(4)	0.062(4)
	0.080(3)	0.131(4)	0.068(3)	-0.004(3)	-0.013(2)	0.063(3)
	0.063(3)	0.104(4)	0.068(3)	-0.018(3)	-0.013(2)	0.043(3)
	0.064(3)	0.093(4)	0.123(4)	-0.030(3)	-0.005(3)	0.025(3)
	0.121(5)	0.185(7)	0.069(3)	-0.032(4)	-0.017(3)	0.091(5)
	0.062(2)	0.064(2)	0.057(2)	-0.001(2)	0.011(2)	0.029(2)
	0.057(2)	0.065(3)	0.069(3)	-0.013(2)	-0.001(2)	0.026(2)
	0.077(3)	0.106(4)	0.092(4)	-0.013(3)	0.012(3)	0.047(3)
	0.074(3)	0.145(6)	0.105(4)	-0.043(4)	0.006(3)	0.059(4)
	0.083(4)	0.111(5)	0.153(6)	-0.039(5)	-0.018(4)	0.057(4)
	0.088(4)	0.093(4)	0.151(6)	0.006(4)	-0.004(4)	0.053(3)
	0.071(3)	0.086(3)	0.110(4)	0.000(3)	0.007(3)	0.044(3)

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The Temperature Factor has the Form of Exp(-T) Where
T = 8*(Pi**2)*U*(Sin(Theta)/Lambda)**2 for Isotropic Atoms
T = 2*(Pi**2)*Sumij(h(i)*h(j)*U(i,j)*Astar(i)*Astar(j)), for
nisotropic Atoms. Astar(i) are Reciprocal Axial Lengths and
h(i) are the Reflection Indices.

Table S5 - Bond Distances (Angstrom)
for: S886B

P1	-O1	1.473(3)	C17	-C18	1.350(12)
P1	-O2	1.577(4)	C18	-C19	1.377(10)
P1	-O3	1.596(3)	C1	-H1	0.930(18)
P1	-N1	1.597(5)	C2	-H2	0.930(16)
O2	-C9	1.458(5)	C3	-H3	0.93(2)
O3	-C13	1.462(5)	C4	-H4	0.930(14)
N1	-C7	1.449(6)	C5	-H5	0.930(11)
N1	-H1A	0.93(7)	C7	-H7	0.980(6)
C1	-C2	1.43(3)	C8	-H8A	0.96(2)
C1	-C6	1.291(11)	C8	-H8B	0.96(3)
C2	-C3	1.31(2)	C8	-H8C	0.96(4)
C3	-C4	1.325(17)	C9	-H9A	0.969(9)
C4	-C5	1.377(13)	C9	-H9B	0.970(9)
C5	-C6	1.363(10)	C11	-H11A	0.96(2)
C6	-C7	1.518(10)	C11	-H11B	0.96(2)
C7	-C8	1.538(10)	C11	-H11C	0.960(9)
C9	-C10	1.524(9)	C12	-H12A	0.96(3)
C10	-C12	1.526(7)	C12	-H12B	0.960(12)
C10	-C13	1.522(8)	C12	-H12C	0.96(5)
C10	-C11	1.518(8)	C13	-H13	0.980(6)
C13	-C14	1.502(7)	C15	-H15	0.930(9)
C14	-C19	1.364(8)	C16	-H16	0.930(10)
C14	-C15	1.389(8)	C17	-H17	0.930(12)
C15	-C16	1.402(10)	C18	-H18	0.930(10)
C16	-C17	1.335(11)	C19	-H19	0.930(9)

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Table S6 - Bond Angles (Degrees) for: S886B

-P1	-O2	113.5(2)	C11	-C10	-C12	110.7(5)
-P1	-O3	114.8(2)	O3	-C13	-C10	110.5(4)
-P1	-N1	113.2(2)	O3	-C13	-C14	106.4(4)
-P1	-O3	101.88(18)	C10	-C13	-C14	115.1(4)
-P1	-N1	106.2(2)	C13	-C14	-C15	120.4(4)
-P1	-N1	106.3(2)	C15	-C14	-C19	116.5(5)
-O2	-C9	114.2(4)	C13	-C14	-C19	123.1(5)
-O3	-C13	118.2(3)	C14	-C15	-C16	120.6(6)
-N1	-C7	126.1(3)	C15	-C16	-C17	120.2(7)
-N1	-H1A	112(4)	C16	-C17	-C18	120.3(8)
-N1	-H1A	122(4)	C17	-C18	-C19	119.9(7)
-C1	-C6	121.6(11)	C14	-C19	-C18	122.4(6)
-C2	-C3	119.0(11)	C2	-C1	-H1	119.3(13)
-C3	-C4	119.5(15)	C6	-C1	-H1	119.1(16)
-C4	-C5	119.6(10)	C1	-C2	-H2	121(2)
-C5	-C6	121.3(8)	C3	-C2	-H2	121(2)
-C6	-C7	120.2(8)	C2	-C3	-H3	120.3(16)
-C6	-C5	117.4(9)	C4	-C3	-H3	120.2(15)
-C6	-C7	122.3(5)	C3	-C4	-H4	120.2(13)
-C7	-C8	109.0(5)	C5	-C4	-H4	120.1(12)
-C7	-C8	111.7(4)	C4	-C5	-H5	119.3(10)
-C7	-C6	111.5(5)	C6	-C5	-H5	119.4(10)
-C9	-C10	113.2(4)	N1	-C7	-H7	108.1(5)
-C10	-C12	106.2(5)	C6	-C7	-H7	108.2(6)
-C10	-C11	109.6(5)	C8	-C7	-H7	108.2(7)
-C10	-C13	112.4(4)	C7	-C8	-H8A	110(2)
-C10	-C13	109.8(5)	C7	-C8	-H8B	109.5(16)
-C10	-C13	107.9(4)	C7	-C8	-H8C	109.5(17)

Table S6 - Bond Angles (Degrees) (continued) for: S886B

H8A	-C8	-H8B	109(3)	H12A	-C12	-H12B	109(3)
H8A	-C8	-H8C	110(4)	H12A	-C12	-H12C	109(3)
H8B	-C8	-H8C	110(3)	H12B	-C12	-H12C	110(4)
O2	-C9	-H9A	109.0(6)	O3	-C13	-H13	108.2(4)
O2	-C9	-H9B	108.9(6)	C10	-C13	-H13	108.2(5)
C10	-C9	-H9A	109.0(6)	C14	-C13	-H13	108.3(6)
C10	-C9	-H9B	108.9(7)	C14	-C15	-H15	119.7(8)
H9A	-C9	-H9B	107.8(8)	C16	-C15	-H15	119.6(8)
C10	-C11	-H11A	109.4(13)	C15	-C16	-H16	119.9(8)
C10	-C11	-H11B	109.5(13)	C17	-C16	-H16	119.9(9)
C10	-C11	-H11C	109.5(7)	C16	-C17	-H17	119.9(10)
H11A	-C11	-H11B	109(2)	C18	-C17	-H17	119.8(10)
H11A	-C11	-H11C	110(3)	C17	-C18	-H18	120.1(9)
H11B	-C11	-H11C	109(3)	C19	-C18	-H18	120.0(9)
C10	-C12	-H12A	109.5(10)	C14	-C19	-H19	118.8(7)
C10	-C12	-H12B	109.4(8)	C18	-C19	-H19	118.8(7)
C10	-C12	-H12C	109.5(11)				

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S2106B : Aug 12 09:18:24 1999

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S U P P L E M E N T A R Y M A T E R I A L
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B E L O N G I N G T O T H E P A P E R

The Formation of Pyrophosphates; An NMR Study on the Mechanism and Stereochemistry of Pyrophosphate Formation from Chiral Dioxaphosphorinanes

b y

Ron Hulst, Johanna M. Visser, N. Koen de Vries, Robert W.J. Zijlstra, Huub Kooijman, Wilberth J.J. Smeets, Anthony L. Spek and Ben L. Feringa

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Table S1 - Crystal Data and Details of the Structure Determination for: S2106B

Crystal Data			
Empirical Formula			C11 H13 C12 O3 P
Formula Weight			295.08
Crystal System			Orthorhombic
Space group		P212121	(No. 19)
a, b, c [Angstrom]	6.4383(10)	10.498(2)	19.571(3)
V [Ang**3]			1322.8(4)
Z			4
D(calc) [g/cm**3]			1.482
F(000)			608
Mu(MoKa) [/mm]			0.6
Crystal Size [mm]		0.15 x 0.15 x	0.25
Data Collection			
Temperature (K)			150
Radiation [Angstrom]	MoKa (graphite monochromator)		0.71073
Theta Min-Max [Deg]			1.6, 27.5
Refined mosaicity (deg)			0.348(1)
Dataset		-8: 8 ; -13: 13 ; -25: 25	
Tot., Uniq. Data, R(int)		35198, 3040,	0.077
Refinement			
wR2, R1, S	0.0590, 0.0228 for 2941 Fo > 4sigma(Fo),		1.059
Npar, w**(-1)	206, sigma**2(F**2)+(0.0323*P)**2+0.21P		
Max. and Av. Shift/Error			0.001, 0.000
Min. and Max. resd. dens. [e/Ang^3]			-0.32, 0.18

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Table S2 - Final Coordinates and Equivalent Isotropic Displacement Parameters of the non-Hydrogen atoms
for: S2106B

Atom	x	y	z	U(eq) [Ang^2]
C17	0.05830(6)	1.17884(3)	0.98672(2)	0.0296(1)
C115	0.49021(7)	1.09130(4)	0.77252(2)	0.0414(1)
P1	-0.10188(5)	1.02163(3)	0.96298(2)	0.0204(1)
O2	-0.06812(15)	1.00339(9)	0.88419(5)	0.0219(3)
O6	0.01990(16)	0.91077(9)	0.99864(4)	0.0237(3)
O8	-0.31824(16)	1.02975(11)	0.98338(6)	0.0301(3)
C3	0.1407(2)	0.97077(14)	0.85862(7)	0.0194(3)
C4	0.2181(2)	0.84918(13)	0.89472(6)	0.0197(4)
C5	0.2257(2)	0.87695(14)	0.97171(7)	0.0230(4)
C9	0.1243(2)	0.96049(14)	0.78178(7)	0.0239(4)
C10	-0.0439(3)	0.89939(15)	0.75146(7)	0.0297(4)
C11	-0.0539(3)	0.88149(17)	0.68108(8)	0.0390(5)
C12	0.1069(4)	0.92444(18)	0.64040(8)	0.0424(6)
C13	0.2735(3)	0.98692(18)	0.66858(8)	0.0402(5)
C14	0.2802(3)	1.00619(16)	0.73929(7)	0.0293(4)
C16	0.0824(3)	0.73241(14)	0.88070(8)	0.0269(4)
C17	0.4426(2)	0.82142(15)	0.87307(7)	0.0255(4)

U(eq) = 1/3 of the trace of the orthogonalized U Tensor

Table S3 - Hydrogen Atom Positions and Isotropic Displacement Parameters
for: S2106B

Atom	x	y	z	U(iso) [Ang^2]
H3	0.225(2)	1.0376(15)	0.8707(7)	0.014(4)
H5A	0.321(3)	0.9480(17)	0.9811(9)	0.023(4)
H5B	0.263(3)	0.8029(18)	0.9961(8)	0.034(5)
H10	-0.163(3)	0.8764(19)	0.7829(10)	0.041(5)
H11	-0.175(3)	0.8428(19)	0.6637(10)	0.036(5)
H12	0.108(3)	0.911(2)	0.5955(11)	0.047(6)
H13	0.394(4)	1.016(2)	0.6454(11)	0.054(6)
H16A	0.096(3)	0.7091(18)	0.8312(10)	0.036(5)
H16B	-0.076(3)	0.7568(18)	0.8909(8)	0.031(4)
H16C	0.126(3)	0.6637(19)	0.9089(9)	0.037(5)
H17A	0.450(3)	0.8025(18)	0.8238(9)	0.036(5)
H17B	0.536(3)	0.8931(16)	0.8843(8)	0.024(4)
H17C	0.495(3)	0.7465(19)	0.8965(9)	0.034(5)

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The Temperature Factor has the Form of Exp(-T) Where
T = 8*(Pi**2)*U*(Sin(Theta)/Lambda)**2 for Isotropic Atoms

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e S4 - (An)isotropic Displacement Parameters
for: S2106B

U(1,1) or U	U(2,2)	U(3,3)	U(2,3)	U(1,3)	U(1,2)
0.0296(2)	0.0238(2)	0.0354(2)	-0.0052(1)	-0.0027(1)	-0.0047(1)
0.0358(2)	0.0459(3)	0.0425(2)	0.0147(2)	0.0091(2)	-0.0092(2)
0.0189(2)	0.0224(2)	0.0200(2)	-0.0029(1)	0.0024(1)	-0.0016(1)
0.0176(4)	0.0280(5)	0.0200(4)	-0.0008(4)	0.0009(3)	0.0032(4)
0.0274(5)	0.0247(5)	0.0189(4)	0.0019(4)	0.0047(4)	0.0005(4)
0.0201(5)	0.0382(6)	0.0320(5)	-0.0103(5)	0.0068(4)	-0.0024(5)
0.0167(6)	0.0228(6)	0.0188(6)	0.0004(5)	0.0025(5)	0.0009(5)
0.0204(7)	0.0204(6)	0.0182(6)	0.0004(5)	0.0002(5)	0.0005(5)
0.0244(7)	0.0245(7)	0.0202(6)	0.0018(5)	-0.0007(5)	0.0030(6)
0.0266(7)	0.0266(7)	0.0185(6)	0.0038(5)	0.0014(5)	0.0071(6)
0.0310(8)	0.0364(8)	0.0218(6)	-0.0017(6)	-0.0018(6)	0.0039(7)
0.0513(11)	0.0409(9)	0.0249(7)	-0.0046(7)	-0.0094(7)	0.0070(8)
0.0718(13)	0.0387(9)	0.0167(7)	0.0003(6)	0.0019(8)	0.0141(9)
0.0584(11)	0.0367(9)	0.0255(8)	0.0093(7)	0.0163(8)	0.0114(8)
0.0336(8)	0.0288(7)	0.0256(7)	0.0077(6)	0.0063(6)	0.0052(6)
0.0333(8)	0.0219(6)	0.0255(7)	0.0000(5)	0.0008(6)	-0.0039(6)
0.0225(7)	0.0287(7)	0.0254(7)	0.0006(6)	0.0005(5)	0.0068(6)

==========

The Temperature Factor has the Form of Exp(-T) Where
T = 8*(Pi**2)*U*(Sin(Theta)/Lambda)**2 for Isotropic Atoms
T = 2*(Pi**2)*Sumij(h(i)*h(j)*U(i,j)*Astar(i)*Astar(j)), for
isotropic Atoms. Astar(i) are Reciprocal Axial Lengths and
h(i) are the Reflection Indices.

Table S5 - Bond Distances (Angstrom)
for: S2106B

Cl7	-P1	2.0008(6)	C12	-C13	1.373(3)
Cl15	-C14	1.7463(19)	C13	-C14	1.399(2)
P1	-O2	1.5689(11)	C3	-H3	0.918(15)
P1	-O6	1.5673(10)	C5	-H5A	0.983(18)
P1	-O8	1.4516(11)	C5	-H5B	0.943(18)
O2	-C3	1.4749(16)	C10	-H10	1.01(2)
O6	-C5	1.4695(17)	C11	-H11	0.94(2)
C3	-C4	1.542(2)	C12	-H12	0.89(2)
C3	-C9	1.511(2)	C13	-H13	0.95(2)
C4	-C5	1.5355(18)	C16	-H16A	1.003(19)
C4	-C16	1.530(2)	C16	-H16B	1.070(19)
C4	-C17	1.5342(19)	C16	-H16C	0.951(19)
C9	-C10	1.391(2)	C17	-H17A	0.986(18)
C9	-C14	1.389(2)	C17	-H17B	0.988(18)
C10	-C11	1.392(2)	C17	-H17C	0.971(19)
C11	-C12	1.382(3)			

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Table S6 - Bond Angles (Degrees)
for: S2106B

C17	-P1	-O2	104.92(4)	O2	-C3	-H3	105.9(9)
C17	-P1	-O6	104.55(4)	C4	-C3	-H3	108.9(9)
C17	-P1	-O8	112.48(5)	C9	-C3	-H3	110.6(9)
O2	-P1	-O6	106.13(5)	O6	-C5	-H5A	108.2(11)
O2	-P1	-O8	114.23(6)	O6	-C5	-H5B	104.3(11)
O6	-P1	-O8	113.65(6)	C4	-C5	-H5A	110.3(10)
P1	-O2	-C3	119.22(8)	C4	-C5	-H5B	110.4(10)
P1	-O6	-C5	118.08(8)	H5A	-C5	-H5B	111.8(16)
O2	-C3	-C4	109.35(10)	C9	-C10	-H10	116.1(11)
O2	-C3	-C9	106.89(10)	C11	-C10	-H10	122.3(11)
C4	-C3	-C9	114.80(12)	C10	-C11	-H11	117.0(12)
C3	-C4	-C5	107.63(11)	C12	-C11	-H11	123.5(12)
C3	-C4	-C16	113.36(11)	C11	-C12	-H12	121.5(13)
C3	-C4	-C17	109.59(11)	C13	-C12	-H12	117.8(13)
C5	-C4	-C16	110.26(11)	C12	-C13	-H13	126.9(13)
C5	-C4	-C17	106.08(10)	C14	-C13	-H13	113.6(14)
C16	-C4	-C17	109.65(12)	C4	-C16	-H16A	108.6(11)
O6	-C5	-C4	111.66(10)	C4	-C16	-H16B	108.6(10)
C3	-C9	-C10	120.77(12)	C4	-C16	-H16C	109.6(12)
C3	-C9	-C14	121.37(12)	H16A	-C16	-H16B	108.8(14)
C10	-C9	-C14	117.80(13)	H16A	-C16	-H16C	110.5(16)
C9	-C10	-C11	121.35(16)	H16B	-C16	-H16C	110.8(15)
C10	-C11	-C12	119.45(17)	C4	-C17	-H17A	110.7(11)
C11	-C12	-C13	120.65(15)	C4	-C17	-H17B	111.5(11)
C12	-C13	-C14	119.37(17)	C4	-C17	-H17C	110.5(11)
C115	-C14	-C9	120.88(11)	H17A	-C17	-H17B	110.0(14)
C115	-C14	-C13	117.79(14)	H17A	-C17	-H17C	106.4(15)
C9	-C14	-C13	121.33(16)	H17B	-C17	-H17C	107.5(15)

Sub 12.