



University of Groningen

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

Hulst, R; Visser, J.M.; De Vries, N.K.; Zijlstra, R.W J; Kooijman, H.; Smeets, W.J.J.; Spek, A.L.; Feringa, B.L.

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Data Collection

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

SUPPLEMENTARY MATERIAL SUPPLEMENTARY MATERIAL ====================================	the Formation of Pyrophosphates; An NMR Study on the Mechanism and Stereochemistry of Pyrophosphate Formation from Chiral Dioxaphosphorinanes	×α	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	Contents	Table S1 - Crystal Data and Details of the Structure Determination for: S691A	Table S2 - Final Coordinates and Equivalent Isotropic Displacement Parameters of the non-Hydrogen atoms for: S691A	Table S3 - Hydrogen Atom Positions and Isotropic Displacement Parameters for: \$691A	Table S4 - (An)isotropic Displacement Parameters for: S691A	Table S5 - Bond Distances (Angstrom) for: S691A	Table S6 - Bond Angles (Degrees) for: S691A
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Crystal Data	Empirical Formula C20 H25 O4 P	Formula Weight 360.37	Crystal System · Orthorhombic	Space group (No. 19)	a, b, c [Angstrom] 6.4799(10) 17.165(2) 17.189(2)	V [Ang**3]	7	D(calc) [g/cm**3] 1.252	768	Mu(MoKa) [/mm] 0.2	
	Empirical Formula	Formula Weight	Crystal System	Space group	a, b, c [Angstrom	V [Ang**3]	8	D(calc) [g/cm**3]	F(000)	Mu (MoKa) [/mm]	

Temperature (K)	295
Radiation [Angstrom]	Moka (Ni filter) 0.71073
Scan, (Type & Range) [Deg]	[Deg] Omega / 2 Theta, 0.66 + 0.35 Tan(Theta)
Hor. and vert. aperture [mm]	ure [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	0.1001, 0.0511 [for 1061 F > 4 sigma(F)], 0.923 $^{\circ}$
w [Sigma**2(F	[sigma**2(F)+(0.0416P)**2]**-1, P=(Max(Fo**2,0)+2Fc**2)/3

0.000, 0.000 -0.22, 0.19

Min. and Max. resd. dens. $[e/Ang^3]$

Max. and Av. Shift/Error

0.1210 0.0930 0.1190 0.1210 0.1130

0.7368(19)

0.3404(12)

0.704(5)

H143

1.062(7)

H161 H171 H181

> 0.078(3) 0.099(4) 0.101(4) 0.095(4)

0.7527(4)

0.9432(13)

C16

0.1802(3) 0.1722(4) 0.1087(5)

0.7516(11)

C15

0.081(3)

0.6624(3) 0.6909(3) 0.7176(3)

0.2459(3)

0.7085(10)

0.8088(10)

0.2312(3)

0.7474(9)

0.6566(10)

C11 C11 C12 C13

65

0.082(3) 0.073(3) 0.060(2) 0.056(2) 0.058(2)

0.5272(4)
0.6000(5)
0.6634(4)
0.6526(3)

0.0520(17)

0.0420(19)

0.5778(3)

0.5697(3)

-0.0116(3) -0.0918(3) -0.1345(3) -0.2061(3) -0.2346(4)

0.8677(8)

0.7746(8)

0.7020(8)

27

0.6102(9)

0.057(2) 0.044(2) 0.061(2) 0.067(2)

0.5529(3)

0.0766(3)

1.1668(9) 1.1047(8) 1.1708(9) 1.2102(8)

0.7667(5)

0.1169(2)

0.5565(3) 0.4824(3) 0.6278(3)

-0.0098(3) -0.0513(3) -0.0436(3)

25 24 25

0.7434(6)

0.843(2)

0.1042(6)

1.117(10) 0.870(3) 0.7852(8)

0.009(3)

0.570(8)

H191

0.7102(14)

0.1287(5)

0.483(8)

H201

Table S3 - Hydrogen Atom Positions and Isotropic Displacement 0.0700 U(iso) [Ang^2] 0.1210 0.1210 0.0750 0.0980 0.0870 0.0720 0.0750 0.0910 0.0620 0.0800 0.0910 0.1010 0.0520 0.0770 0.0770 0.0910 0.1010 0.1010 0.5437 (14) 0.6536(5) 0.6513(16) 0.702(2) 0.6080(7) 0.713(3) 0.697(2) 0.5883(3) 0.484(3) 0.467(2) 0.4752(10)0.6300(11) 0.613(2) 0.4865(8) 0.6765(14) 0.6246(10) 0.5997(18) 0.5465(3) 0.4365(13) N 0.3255(7) 0.3645(12) 0.2738(14) 0.2483(3) -0.2355(18) -0.0903(15)0.2317(3) -0.0334(17) -0.287(3)-0.2141(12) 0.1005(9) 0.0803(3) -0.0452(15)-0.1021(17) 0.0165(13) -0.1156(10) -0.0278(14)-0.1084(16)-0.0181(16) S691A 0.923(5) Parameters 0.969(5)0.543(8)0.753(6)1.184(4) 0.7144(10) 0.565(3) 0.6444(12)0.792(2)0.7648(16) 1.365(5) 0.8376(16) 0.513(4) 1.324(4) 1.152(4) 1.136(4) 1.096(4) 1.1328(14) 1.309(5)for: H142 H131 H141 H101 H111 H121 H122 Atom H71. H81 H42 H91 H43 H51 H33 H41 H11 H12 H32 H31

- Final Coordinates and Equivalent Isotropic Displacement

Parameters of the non-Hydrogen atoms

S691A

for:

tom

able s2

U(eq) [Ang^2]

N

0.0458(5)

0.48444(9)

0.11286(8) 0.1396(2) 0.1547(2)

0.8230(2)

0.7471(7) 0.7368(6) 1.0648(5)

0.0683(14) 0.0503(14) 0.0553(12) 0.0420(12)

0.4101(2) 0.5582(2) 0.4897(2) 0.5023(2) The Temperature Factor has the Form of Exp(-T) Where T = 8*(Pi**2)*U*(Sin(Theta)/Lambda)**2 for Isotropic Atoms

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

0.7314(3)

0.1241(4)

0.6058(11)

C20

0.8119(4)

0.0519(5)

0.8395(18)

C18 C19 7 g

P1 -01 1.444 (4) C19 -C20 1.391 (10) P1 -02 1.561 (4) C1 +H1 0.93 (3) P1 -03 1.571 (4) C1 +H1 0.93 (3) P1 -04 1.569 (4) C3 +H3 1.01 (2) 02 -C12 1.459 (6) C3 -H31 1.01 (3) 03 -C11 1.448 (6) C3 -H32 1.01 (3) 03 -C12 1.459 (6) C3 -H31 1.01 (3) 04 -C5 1.448 (6) C3 -H31 1.01 (3) C2 -C3 1.448 (6) C3 -H41 1.01 (3) C2 -C3 1.538 (7) C4 -H42 1.01 (3) C2 -C4 1.539 (7) C4 -H41 1.02 (3) C3 -C4 1.339 (7) C5 -H51 1.01 (3) C4 -C4 -H41 1.02 (3) 1.02 (3) C5 -C6 <th>rable</th> <th>55</th> <th>Bond Distances (Ang for: S691A</th> <th>(Angstrom)</th> <th></th> <th></th>	rable	55	Bond Distances (Ang for: S691A	(Angstrom)		
-02 1.561(4) C1 -H11 0.93(-03 1.571(4) C1 -H12 0.93(-04 1.569(4) C3 -H31 0.93(-C12 1.459(6) C3 -H32 1.01(-C12 1.448(6) C3 -H32 1.01(-C2 1.448(6) C3 -H32 1.01(-C3 1.448(6) C3 -H32 1.01(-C4 1.448(6) C3 -H32 1.01(-C3 1.477(6) C4 -H42 1.01(-C4 1.538(7) C4 -H42 1.02(-C4 1.551(7) C4 -H42 1.02(-C4 1.538(7) C7 -H31 0.94(-C5 1.538(7) C1 -H101 0.94(-C6 1.389(7) C1 -H101 0.94(-C7 1.398(7) C1 -H101 0.94(-C8 1.389(7) C1 <td>P1</td> <td>-01</td> <td></td> <td>C19</td> <td>-C20</td> <td>1.391(10)</td>	P1	-01		C19	-C20	1.391(10)
-03 1.571(4) C1 -H12 0.93(4) -04 1.569(4) C3 -H31 1.01(4) -C12 1.459(6) C3 -H32 1.01(4) -C1 1.448(6) C3 -H32 1.01(4) -C2 1.448(6) C3 -H32 1.01(4) -C3 1.448(6) C3 -H33 1.01(4) -C2 1.448(6) C3 -H31 1.01(6) -C3 1.448(6) C4 -H41 1.02(6) -C3 1.538(7) C4 -H42 1.02(6) -C4 1.519(7) C4 -H42 1.02(6) -C4 1.519(7) C4 -H41 0.90(6) -C5 1.538(7) C7 -H31 0.94(6) -C6 1.380(7) C10 -H11 0.94(6) -C7 1.394(9) C10 -H12 1.04(6) -C11 1.394(1) C12 -H12 1.04 -C12 1.39	P1	-03		ប	-H11	0.93(3)
-04 1.569 (4) C3 -H31 1.01 -C12 1.459 (6) C3 -H32 1.01 -C1 1.448 (6) C3 -H32 1.01 -C5 1.448 (6) C3 -H33 1.01 -C5 1.477 (6) C4 -H41 1.01 -C2 1.538 (7) C4 -H42 1.02 (-C3 1.519 (7) C4 -H43 1.02 (-C4 1.519 (7) C4 -H43 1.02 (-C5 1.553 (7) C4 -H43 1.02 (-C6 1.559 (7) C4 -H43 1.02 (-C7 1.379 (8) C9 -H91 1.02 (-C7 1.379 (1) C1 -H101 0.99 (-C1 1.378 (11) C12 -H121 1.04 -C1 1.394 (9) C12 -H122 1.04 -C1 1.394 (9) C13 -H121 0.96 -C1 1.390 (8)	P1	-03	•	CJ	-H12	0.93(3)
-C12 1.459 (6) C3 -H32 1.01 (-C1 1.448 (6) C3 -H32 1.01 (-C5 1.448 (6) C4 -H41 1.02 (-C2 1.538 (7) C4 -H42 1.02 (-C3 1.513 (7) C4 -H42 1.02 (-C4 1.519 (7) C4 -H43 1.02 (-C4 1.519 (7) C4 -H43 1.02 (-C4 1.519 (7) C4 -H43 1.02 (-C5 1.519 (7) C5 -H51 0.91 (-C6 1.519 (7) C7 -H71 0.91 (-C7 1.379 (8) C9 -H91 1.02 (-C7 1.389 (7) C10 -H111 0.95 (-C9 1.389 (7) C11 -H121 1.04 (-C9 1.394 (9) C13 -H121 0.96 (13 -C14 1.540 (8) C14 -H142 0.96 (<t< td=""><td>P1</td><td>-04</td><td></td><td>33</td><td>-H31</td><td>1.01(2)</td></t<>	P1	-04		33	-H31	1.01(2)
-C1 1.448 (6) C3 -H33 1.01 -C5 1.477 (6) C4 -H41 1.02 (1) -C2 1.538 (7) C4 -H42 1.02 (1) -C3 1.518 (7) C4 -H43 1.02 (1) -C4 1.519 (7) C4 -H43 1.02 (1) -C5 1.553 (7) C4 -H43 1.02 (1) -C6 1.553 (7) C7 -H71 0.90 (1) -C6 1.553 (7) C7 -H31 0.90 (1) -C7 1.379 (8) C9 -H91 0.90 (1) -C7 1.380 (7) C10 -H111 0.90 (1) -C8 1.380 (7) C11 -H121 1.04 (1) -C9 1.380 (7) C11 -H121 1.04 (1) -C9 1.378 (11) C12 -H121 1.04 (1) -C1 1.394 (9) C13 -H141 0.95 -C13 1.556 (8) C14 -H142 0.95	02	-C12		C3	-н32	1.01(3)
-CS 1.477 (6) C4 -H41 1.02 (-C2 1.538 (7) C4 -H42 1.02 (-C3 1.521 (7) C4 -H43 1.02 (-C4 1.519 (7) C5 -H51 0.91 (-C5 1.553 (7) C7 -H71 0.90 (-C6 1.553 (7) C8 -H91 0.94 (-C7 1.379 (8) C9 -H91 1.02 (-C7 1.379 (8) C9 -H91 1.02 (-C7 1.380 (7) C10 -H101 0.94 (-C8 1.380 (7) C10 -H111 0.95 (-C9 1.380 (7) C11 -H121 1.04 -C9 1.353 (11) C12 -H121 1.04 -C10 1.358 (11) C12 -H121 1.04 -C11 1.526 (8) C14 -H143 0.95 -C12 1.387 (11) C16 -H161 0.95 -C20	03	-C1		C3	-н33	1.01(3)
-C2 1.538(7) C4 -H42 1.02 (-C3 1.521 (7) C4 -H43 1.02 (-C4 1.519 (7) C5 -H51 0.91 (-C5 1.553 (7) C5 -H51 0.90 (-C6 1.553 (7) C7 -H71 0.90 (-C6 1.553 (7) C8 -H81 0.90 (-C7 1.379 (8) C9 -H91 0.94 (-C7 1.380 (7) C10 -H101 0.95 (-C8 1.380 (7) C11 -H111 0.95 (-C9 1.338 (11) C12 -H121 1.04 (-C9 1.378 (11) C12 -H121 1.04 (-C10 1.378 (11) C12 -H121 1.04 (-C11 1.540 (8) C14 -H141 0.96 (-C15 1.387 (11) C16 -H141 0.95 (-C20 1.378 (11) C16 -H161 1.10 -C20 </td <td>04</td> <td>-C5</td> <td></td> <td>C4</td> <td>-H41</td> <td>1.02(3)</td>	04	-C5		C4	-H41	1.02(3)
-C3 1.521(7) C4 -H43 1.02(7) -C4 1.519(7) C5 -H51 0.91(8) -C5 1.553(7) C7 -H71 0.90(8) -C6 1.509(7) C8 -H81 0.94(8) -C7 1.379(8) C9 -H81 0.94(8) -C7 1.379(8) C9 -H91 1.02(8) -C8 1.380(7) C10 -H101 0.94(8) -C9 1.378(11) C12 -H121 1.04(8) -C11 1.378(11) C12 -H121 1.04(8) -C12 1.540(8) C14 -H142 0.96 -C13 1.500(7) C14 -H143 0.96 -C15 1.387(11) C16 -H144 0.96 -C26 1.378(11) C16 -H141 0.96 -C27 1.378(11) C16 -H141 0.96 -C29 1.372(13) C19 -H141 0.96 -C29	C	-C2		C4	-H42	1.02(3)
-C4 1.519 (7) C5 -H51 0.91 (91) (-C5 1.553 (7) C7 -H51 0.90 (91) (-C5 1.509 (7) C7 -H71 0.90 (91) (-C6 1.509 (7) C8 -H81 0.94 (91) (-C1 1.389 (7) C10 -H101 0.93 (91) (-C1 1.389 (7) C11 -H111 0.93 (91) (-C2 1.353 (11) C12 -H121 1.04 (91) (-C3 1.353 (11) C12 -H121 1.04 (91) (-C1 1.394 (9) C13 -H121 1.04 (91) (-C1 1.394 (9) C13 -H121 0.96 (91) (-C1 1.389 (11) C12 -H122 1.04 (91) (-C1 1.389 (11) C14 -H142 0.96 (91) (-C1 1.389 (11) C14 -H142 0.96 (91) (-C1 1.378 (11) C18 -H161 1.10 (91) (-C1 1.378 (11) C18 -H181 0.93 (11) (-C1 1.372 (13) C19 -H191 0.72 (13) (-C1 1.373 (14) C20 -H201 0.88	C2	-63		C4	-H43	1.02(3)
-C5 1.553 (7) C7 -H71 0.90(-C6 1.509 (7) C8 -H81 0.94 (-C7 1.379 (8) C9 -H91 1.02 (-C11 1.389 (7) C10 -H101 0.93 (-C8 1.380 (7) C11 -H101 0.93 (-C9 1.380 (7) C11 -H111 0.93 (-C9 1.378 (11) C12 -H121 1.04 (-C10 1.378 (11) C12 -H141 0.96 3 -C14 1.540 (8) C14 -H142 0.96 3 -C14 1.540 (8) C14 -H143 0.96 5 -C16 1.387 (11) C16 -H143 0.96 5 -C20 1.376 (9) C17 -H161 0.93 6 -C17 1.372 (13) C19 -H181 0.96 6 -C18 1.373 (14) C20 -H201 0.93	C2	-C4		C5	H51	0.91(3)
-C6 1.509 (7) C8 -H81 0.94 (6) -C7 1.379 (8) C9 -H91 1.02 (6) -C11 1.389 (7) C10 -H101 0.93 (6) -C8 1.380 (7) C11 -H111 0.93 (6) -C9 1.353 (11) C12 -H121 1.04 (6) -C10 1.378 (11) C12 -H121 1.04 (7) 2 -C13 1.526 (8) C14 -H141 0.96 3 -C14 1.540 (8) C14 -H142 0.96 3 -C15 1.500 (7) C14 -H143 0.96 5 -C20 1.370 (9) C17 -H141 0.96 6 -C17 1.378 (11) C18 -H161 0.95 7 -C18 1.372 (13) C19 -H191 0.95 8 -C19 1.373 (14) C20 -H201 0.88	C3	50-	•	C7	-H71	0.90(3)
-C7 1.379(8) C9 -H91 1.02 -C11 1.389(7) C10 -H101 0.93(-C8 1.380(7) C11 -H111 0.97(-C9 1.353(11) C12 -H121 1.04(-C10 1.378(11) C12 -H121 1.08 2 -C11 1.394(9) C13 -H121 1.08 3 -C14 1.526(8) C14 -H141 0.96 3 -C15 1.500(7) C14 -H143 0.96 5 -C16 1.387(11) C16 -H161 1.10 5 -C20 1.378(11) C16 -H161 0.95 6 -C17 1.378(11) C18 -H181 0.95 7 -C18 1.372(13) C19 -H181 0.93 8 -C19 1.373(14) C20 -H201 0.88	CS	90-		82	-H81	0.94(4)
-C11 1.389(7) C10 -H101 0.93(-C8 1.380(7) C11 -H111 0.97(-C9 1.353(11) C12 -H121 1.04(-C10 1.378(11) C12 -H122 1.04(-C11 1.394(9) C13 -H131 1.08(2 -C13 1.526(8) C14 -H141 0.96(3 -C14 1.540(8) C14 -H142 0.96(5 -C16 1.387(11) C16 -H161 1.10(5 -C20 1.377(1) C16 -H161 0.96(6 -C17 1.378(11) C18 -H181 0.95(7 -C18 1.372(13) C19 -H191 0.93(8 -C19 1.373(14) C20 -H201 0.88(90	-C7		60	-н91	1.02(5)
-C8 1.380(7) C11 -H111 0.97 -C9 1.353(11) C12 -H121 1.046 -C10 1.378(11) C12 -H122 1.046 0 -C11 1.394(9) C13 -H131 1.046 2 -C13 1.526(8) C14 -H141 0.96 3 -C14 1.540(8) C14 -H142 0.96 3 -C15 1.500(7) C14 -H143 0.96 5 -C20 1.377(11) C16 -H161 1.10 6 -C17 1.378(11) C18 -H181 0.95 7 -C18 1.372(13) C19 -H191 0.93 8 -C19 1.373(14) C20 -H201 0.88	90	-C11	1.389(7)	C10	-н101	0.93(5)
-C9 1.353(11) C12 -H121 1.04 -C10 1.378(11) C12 -H122 1.04 0 -C11 1.394(9) C13 -H131 1.08 2 -C13 1.526(8) C14 -H141 0.96 3 -C14 1.540(8) C14 -H142 0.96 3 -C15 1.500(7) C14 -H143 0.96 5 -C16 1.387(11) C16 -H161 1,10 6 -C17 1.378(11) C18 -H181 0.95 7 -C18 1.372(13) C19 -H181 0.93 8 -C19 1.373(14) C20 -H201 0.72	C7	. C8	1.380(7)	C11	-н111	0.97(3)
-C10 1.378(11) C12 -H122 1.04 0 -C11 1.394(9) C13 -H131 1.08 2 -C13 1.526(8) C14 -H141 0.96 3 -C14 1.540(8) C14 -H142 0.96 3 -C15 1.500(7) C14 -H143 0.96 5 -C16 1.377(11) C16 -H161 1.10 6 -C17 1.378(11) C18 -H181 0.93 7 -C18 1.372(13) C19 -H191 0.72 8 -C19 1.373(14) C20 -H201 0.88	82	62-		C12	-н121	1.04(2)
-C11 1.394(9) C13 -H131 1.08 -C13 1.526(8) C14 -H141 0.96 -C14 1.540(8) C14 -H142 0.95 -C15 1.500(7) C14 -H143 0.96 -C16 1.387(11) C16 -H161 1.10 -C20 1.370(9) C17 -H171 0.95 -C17 1.378(11) C18 -H181 0.93 -C18 1.372(13) C19 -H191 0.72	60	-C10		C12	-H122	1.04(3)
-C13 1.526(8) C14 -H141 0.96 -C14 1.540(8) C14 -H142 0.95 -C15 1.500(7) C14 -H143 0.96 -C16 1.387(11) C16 -H161 1,10 -C20 1.370(9) C17 -H171 0.95 -C17 1.378(11) C18 -H181 0.93 -C18 1.372(13) C20 -H201 0.88	C10		1.394(9)	C13	-H131	1.08(5)
-C14 1.540(8) C14 -H142 0.95 -C15 1.500(7) C14 -H143 0.96 -C16 1.387(11) C16 -H161 1.10 -C20 1.370(9) C17 -H171 0.95 -C17 1.378(11) C18 -H181 0.93 -C18 1.372(13) C19 -H191 0.72	C12		1.526(8)	C14	-H141	0.96(3)
-C15 1.500(7) C14 -H143 0.96 -C16 1.387(11) C16 -H161 1.10 -C20 1.370(9) C17 -H171 0.95 -C17 1.378(11) C18 -H181 0.93 -C18 1.372(13) C19 -H191 0.72	C13			C14	-H142	0.95(3)
-C16 1.387 (11) C16 -H161 1.10 -C20 1.370 (9) C17 -H171 0.95 -C17 1.372 (13) C19 -H191 0.93 -C18 1.372 (13) C20 -H201 0.88	C13		1.500(7)	C14	-H143	0.96(3)
-C20 1.370(9) C17 -H171 0.95 -C17 1.378(11) C18 -H181 0.93 -C18 1.372(13) C19 -H191 0.72 -C19 1.373(14) C20 -H201 0.88	C15		1:387 (11)	,C16	-H161	1,10(4)
-C17 1.378(11) C18 -H181 0.93 -C18 1.372(13) C19 -H191 0.72 -C19 1.373(14) C20 -H201 0.88	C15		1.370(9)	C17	-H171	6.
-C18 1.372(13) C19 -H191 0.72 -C19 1.373(14) C20 -H201 0.88	C16			, C18	-H181	0.93(5)
-C19 1.373(14) C20 -H201 0.88	C17			C19	-H191	0.72(5)
	C18		1.373 (14)	C20	-H201	0.88(5)

0.004(3) 0.000(3) -0.003(4) 0.013(4) 0.002(4)

-0.001(4)

-0.003(3)

0.017(5)

0.019(5)

0.003(4)

0.047(4)

0.021(5)

0.001(3)

0.010(3)

0.006(3) 0.010(3) 0.014(4) 0.005(3)

0.009(4)

-0.002(3) 0.018(3) 0.002(3)

0.069(4)

0.050(4)

0.044(4)

0.035(4)

0.038(3)

0.078(4)

0.059(4) 0.064(3) 0.064(3) 0.099(5) 0.125(6)

0.044(4) 0.028(3) 0.045(3) 0.054(4) 0.071(5)

-0.007(3)

0.002(3)

-0.003(3)

0.046(3)

-0.003(2)

0.046(2)

0.067(4)
0.067(4)
0.052(4)
0.063(3)
0.080(4)
0.045(3)

0.041(2)

0.050(2)

0.005(2) -0.013(2) -0.004(3) 0.000(3)

0.008(2)

0.0034(8) 0.009(3) 0.005(2) -0.003(2)

-0.0050(8) -0.017(2) 0.000(2)

-0.0016(8)

0.0461(9)

0.0398(8) 0.065(2) 0.039(2) 0.055(2)

0.0516(10)

0.094(3)

0.009(2)

0.046(2) 0.058(2) 0.061(2)

-0.008(2)

U(1,2)

U(1,3)

U(2,3)

U(3,3)

U(1,1) or U U(2,2)

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)

0.007(7)

0.003(7)

0.009(4)

0.131(7)

0.099(7)

0.093(5)

-0.002(4)

-0.009(5)

0.004(4)

-0.016(3)

0.003(3)

0.001(4) 0.009(4) -0.015(5) -0.015(5)

-0.013(3)

0.061(4)

0.054(3)

0.060(4) 0.108(6) 0.070(5) 0.083(6)

0.070(4) 0.042(3) 0.057(4) 0.067(5) 0.042(4)

0.064(4)

0.051(3)

0.002(3)

0.011(4)

0.063(4)

0.067(4)

0.049(4)

0.071(5)

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.

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Displacement Parameters

(An)isotropic

S4

© 20 	00 Ar	ner	ica	n C	hei	mic	al	Soc	ciet	y, .	J. A	m.	Cł	nen	1. S	oc.	., H	luls	st ja	.992	277	'0k	Su	pp	orti	ing	Inf	o F	Page	e 4
		107.6(6)	107.5(7)	109.5(17)	109.5(10)	109.6(15)	109(3)	109(2)	110(3)	119.6(19)	120.1(19)	119.2(17)	119.4(16)	120.5(19)	120.9(19)	120(4)	120(4)	119.3(14)	119.4(15)											
		-H131	-H131	-H141	-H142	-H143	-н142	-H143	-н143	-H161	-H161	-H171	-H171	-н181	-н181	-H191	-H191	-н201	-H201											
	ned)	-C13	-C13	-C14	-C14	-C14	-C14	-C14	-C14	-c16	-C16	-C17	-C17	-C18	-C18	-C19	-C19	-C20	-C20											
	(continued)	C14	C15	C13	C13	C13	H141	H141	H142	C15	C17	C16	C18	C17	C19	C18	C20	C15	C19											,
8	(Degrees)	108.5(16)	108.8(9)	108.8(15)	119.9(13)	119.6(14)	120(2)	120(2)	119.8(10)	119.8(11)	120.2(17)	120.1(17)	119.9(18)	120.3(18)	110.5(13)	110.5(5)	110.5(12)	110.6(6)	108.7(9)	107.7(7)										
	Bond Angles for: S691A	-H51	-H51	-H51	-H71	-H71	-H81	-H81	-н91	-H91	~H101	-H101	-H111	-H111	-н121	-H122	-H121	-H122	-H122	-H131										
ı	1	-C5	-65	-05	-C7	- CJ -	8D-	-C8	60-	60-	-C10	-C10	-C11	-c11	-c12	-C12	-C12	-C12	-c12	-C13										5
	Table S6	04	C2	92	92	C8	ζ2	60	82	C10	ေ	C11	.9 2	C10	05	05	C13	C13	H121	C12										م م
																									,					
·		107.8(5)	111.0(5)	114.9(5)	121.1(6)	120.6(6)	118.2(5)	120.3(7)	121.4(8)	118.6(8)	120.2(8)	121.3(7)	109.3(13)	109.3(6)	109.2(12)	109.3(6)	107.9(9)	109.4(14)	109.5(10)	109.4(12)	109.6(18)	109.6(19)	109(2)	109.4(15)	109.5(12)	109.4(13)	109.6(19)	109(2)	110(2)	
		-C14	-C15	-C15	-C16	-C20	-C20	-C17	-C18	-C19	-C20	-C19	-H11	-H12	-H11	-H12	-H12	-н31	-H32	-н33	-н32	-н33	-н33	-H41	-H42	-H43	-H42	-H43	-H43	
		-C13	-C13	-C13	-C15	-C15	-C15	-C16	-C17	-C18 .	-C19	-C20	-	-C1	,-c1	-C1	-C1	-63	-03	-C3	-C3	-03	ည	-C4	-C4	-C4	-C4	-C4	-C4	,
		C12	C12	C14	C13	C13	C16	C15	C16	C17	C18	C15	03	03	C2	C2	H11	C2	C2	C2	н31	H31	Н32	C2	C2	C2	H41	H41	H42	·
. 7	(Degrees)	116.8(2)	112.1(2)	. 113.4(2)	106.9(2)	101.3(2)	105.23(19)	122.7(3)	118.5(3)	117.7(3)	111.8(4)	110.1(4)	106.4(4)	106.5(4)	111.7(4)	113.1(4)	108.6(4)	108.4(4)	107.0(4)	115.3(4)	123.3(5)	117.5(5)	119.0(5)	120.6(6)	120.5(6)	120.4(6)	119.7(6)	119.8(5)	106.1(4)	
	Bond Angles for: S691A	-02	-03	-04	-03	-04	-04	-C12	-C1	-C5	-C2	-C3	-C4	-C5	-C4	-C5	-C5	-C2	90-	92-	-C7	-C11	-c11	8D-	62-	-C10	-C11	-C10	-c13	
·	e S6 - Bo	P1	-P1	-P1	-P1	-P1	-P1	-02	-03	-04	-C1	-C2	-C2	-c2	-C2	-25	-C2	-C5	-C5	-C5	92-	92-	92-	-C7	-C8	60-	-C10	-C11	-C12	

S886B	

: Aug 12 09:17:35 1999

ERIAL MAA UPPLEMENTARY

PAPER T HE T 0 BELONGING The Formation of Pyrophosphates; An NMR Study on the Mechanism and Stereochemistry of Pyrophosphate Formation from Chiral Dioxaphosphorinanes

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

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Crystal Data and Details of the Structure Determination for: \$888BTable S1

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

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

Displacement Parameters (An)isotropic for: S886B Table S4

Bond Distances (Angstrom) for: S886B Table S5

Bond Angles for: S886B Table S6

- Crystal Data and Details of the Structure Determination Table S1

Crystal Data

Empirical Formula		, C19	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]	06.	06.	120
V [Ang**3]			1457.0(3)
23			е
D(calc) [g/cm**3]			1.181
F(000)			552
Mu(CuKa) [/mm]			1.4
Crystal Size [mm]		0.05 × 0	0.05×0.70

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

Table S3 - Hydrogen Atom Positions and Isotropic Displacement	Parameters	for: S886B
33		
Table		

Table S2 - Final Coordinates and Equivalent Isotropic Displacement

Parameters of the non-Hydrogen atoms for: \$886B

U(eq) [Ang²]

0.0641(4)

0.97802(9)

0.16672(10)

0.07639(10)

Pl

×

Atom

0.0764(12) 0.0628(11) 0.0746(14) 0.235(7)

1.0184(2)

0.2436(3) 0.1962(4) 0.4702(8) 0.5081(10)

0.0108(2)

1.0050(3)

0.0326(3)

0.0045(3)

Atom	×	>	D z	U(iso) [Ana^2]
 H1	0.1936(13)	0.5300(8)	- 7 (12)	10
H1A	0.079(5)	0.134(6)	0.777(6)	0.1120
Н2	0.0744(18)	0.5920(10)	0.6326(16)	0.3520
Н3	-0.1207(11)	0.4449(12)	0.5808(10)	0.1910
H4	-0.1588(7)	0.2473(10)	0.5575(7)	0.1640
HS	-0.0376(6)	0.1863(7)	0.6626(7)	0.1320
Н7	0.2221(5)	0.3789(4)	0.8468(4)	0.0910
нва	0.319(3)	0.4010(16)	0.653(3)	0.1740
н8в	0.2342(7)	0.258(3)	0.631(3)	0.1740
н8С	0.333(3)	0.306(5)	0.7366(9)	0.1740
н9А	0.2729(5)	0.2905(6)	1.2175(4)	0.1070
н9в	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

0.116(3)

0.6922(6)

0.3210(7)

0.2752(6)

0.7813(4)

0.7181(4)

0.6611(7)

0.0778(19)

1.2090(4)

1.1634(6) 1.3481(5) 1.1494(4) 1.1598(4)

1.1802(4)

0.2505(6) 0.3263(5) 0.4586(5)

0.1911(5) 0.1284(4) 0.2092(5)

65

c10

C11 C12 C13 C14

0.119(3)

0.0621(12) 0.0657(16) 0.091(2) 0.106(3) 0.112(3) 0.107(3)

0.2616(4)

0.0004(4)

0.3239(8)

0.1148(6)

1.2580(5) 1.2715(6) 1.1889(8)

0.3074(6)

C15

0.3754(7)

-0.2120(5)

C16

0.3297(4)

-0.0708(4)

0.159(5)

0.6059(10)

0.4231(12) 0.3055(10) 0.2699(7) 0.3548(5) 0.3186(4)

0.6462(16)

0.0463(18)

0.1215(13)

0.7112(12)

0.137(4) 0.110(3) 0.0792(18) 0.0758(16)

0.6011(7)

-0.0897(7) -0.0149(6) 0.0918(6) 0.1780(5) The Temperature Factor has the Form of Exp(-T) Where T = 8*(Pi**2)*U*(Sin(Theta)/Lambda)**2 for Isotropic Atoms

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

1.0770(6)

0.4168(5)

-0.0602(5)

C19

1.0921(7)

0.4844(6).

-0.1196(6)

C18

0.4616(7)

-0.1958(6)

0.96(2)

c11C11

-05 920.96(2) 0.960(9)

-H11B -H11A

-H11C -H12A

C11 C12

> -C10 -C12

-08

-C7

0.970(9)

(6)696.0

0.96(3) 0.960(12) 0.96(5)

-H12B

-H12C

c12C13 C15

-C13

-C11

-C14 -C19 -C15 -C16

0.980(6) 0.930(9)

-H13 -H15 -H16

0.930(10) 0.930(12)

C16 C17 0.930 (10)

-H18

C18C19

-H17

0.930(9)

-H19

-C17-

0.980(6)

-H7

-H1A

-C2 9 2 5 -C4

-C7

0.96(2)

-H8A

0.96(3) 0.96(4)

-H8B -H8C -H9A -H9B

> 85 65 ç

1.377 (10)

-C19

C18

1.577(4) 1.473(3)

-01 -02 -03 -N1 65

1.350 (12)

-C18

Table S5 - Bond Distances (Angstrom)

for: S886B

0.930(16) 0.93(2) 0.930(14)0.930(11)

0.930(18)

-H1 -H2 -H3 -H4 -H5

 \Box $\mathcal{C}_{\mathcal{C}}$ \mathbb{S} c_4 S C_{7} 85 85

1.596(3) 1.597(5) 1.458(5) 1.462(5) 1.449(6) 0.93(7) 1.43(3) 1.291(11) 1.31(2) 1.325(17) 1.377(13) 1.363(10) 1.518(10) 1.538(10) 1.524(9) 1.526(7) 1.522(8) 1.518(8) 1.502(7) 1.364(8) 1.389(8) 1.402(10) 1.335(11)

-C13

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(5) 0.0034(5) 0.0142(5) 0.0353(6)	(2) 0.012(2) 0.035(2) 0.038(2)	(2) 0.000(2) 0.0054(15) 0.050(2)	0.0578(15)-0.0099(13) 0.0000(13) 0.0330(14)	(2) 0.005(2) 0.022(2) 0.032(2)	13) -0.057(6) -0.195(12) 0.107(7)	(2) -0.069(10) -0.24(2) 0.149(11)	(8) 0.010(7) 0.018(7) 0.146(9)	(5) 0.044(5) 0.022(4) 0.084(6)	(4) 0.036(4) 0.010(4) 0.051(4)	(2) -0.004(2) 0.007(3) 0.046(3)	(2) 0.009(2) 0.012(2) 0.032(2)	(5) 0.051(4) 0.050(4) 0.062(4)	(3), -0.004(3) -0.013(2) 0.063(3)	(3) -0.018(3) -0.013(2) 0.043(3)	(4) -0.030(3) -0.005(3) 0.025(3)	(3) -0.032(4) -0.017(3) 0.091(5)	(2) -0.001(2) 0.011(2) 0.029(2)	(3) -0.013(2) -0.001(2) 0.026(2)	(4) -0.013(3) 0.012(3) 0.047(3)	(4) -0.043(4) 0.006(3) 0.059(4)	(6) -0.039(5) -0.018(4) 0.057(4)	(6) 0.006(4) -0.004(4) 0.053(3)	(4) 0.000(3) 0.007(3) 0.044(3)
0.0603(5)	0.082(2)	0.072(2)	0.0578(19	0.065(2)	0.285(13)	0.40(2)	0.175(8)	0.116(5)	0.117(4)	0.059(2)	0.066(2)	0.121(5)	0.068(3)	0.068(3)	0.123(4)	0.069(3)	0.057(2)	0.069(3)	0.092(4)	0.105(4)	0.153(6)	0.151(6)	0.110(4)
0.0632(7)	0.061(2)	0.100(2)	0.070(2)	0.062(2)	0.079(5)	0.102(7)	0.171(9)	0.196(9)	0.103(5)	0.064(3)	0.064(3)	0.134(5)	0.131(4)	0.104(4)	0.093(4)	0.185(7)	0.064(2)	0.065(3)	0.106(4)	0.145(6)	0.111(5)	0.093(4)	0.086(3)
0.0712(7)	0.108(2)	0.068(2)	0.061(2)	0.089(3)	0.343(15)	0.41(2)	0.202(10)	0.109(5)	0.108(4)	0.116(4)	0.089(3)	0.099(4)	0.080(3)	0.063(3)	0.064(3)	0.121(5)	0.062(2)	0.057(2)	0.077(3)	0.074(3)	0.083(4)	0.088(4)	0.071(3)

T = 8*(Pi**2)*(")*(Sin(Theta)/Lambda)**2 for Isotropic Atoms

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

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S4 - (An)isotropic Displacement Parameters

for: \$886B

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© 20	000 Ar	ner	ica	n C	Che	mic	cal	So	ciet	ty, .	J. <i>A</i>	۱m.	. Cl	nen	ı. S	Soc	., H	Iuls	st ja	ເ99	277	70k	Su	ıpp	orti	ing	Inf	o I	Page	8 🦪
-		109(3)	109(3)	110(4)	108.2(4)	108.2(5)	108.3(6)	119.7(8)	119.6(8)	119.9(8)	119.9(9)	119.9(10)	119.8(10)	120.1(9)	120.0(9)	.118.8(7)	118.8(7)			<u>.</u>										
		-н12в	-H12C	-H12C	-H13	-H13	-н13	-H15	-H15	-H16	-H16	-H17	-н17	-H18	-H18	-н19	-H19				-									
	ned)	-C12	-C12	-C12	-C13	-C13	-c13	-c15	-C15	-c16	-C16	-C17	-C17	-C18	-C18	-C19	-C19													
	(continued)	H12A	H12A	Н12В	03	C10	C14	C14 .	C16	C15	C17	C16	C18	C17	C19	C14	C18						i.				٠			
8 -	(Degrees)	109(3)	110(4)	110(3)	109.0(6)	108.9(6)	109.0(6)	108.9(7)	107.8(8)	109.4(13)	109.5(13)	109.5(7)	109 (2)	110(3)	109(3)	109.5(10)	109.4(8)	109.5(11)		`										
	Bond Angles for: \$886B	-H8B	-H8C	-H8C	-н9.	-н9в	-н9д	-н9в	-н9в	-H11A	-H11B	-H11C	 _H11B	-H11C	-H11C	-H12A	-H12B	-H12C												
	S6 - Bor fol	-C8	. 8	82-	6၁-	60-	60-	60-	-C9	-C11	-C11	-C11	-C11	-C11	-c11	-c12	-C12	-C12		·										∞
	Table	нва	нва	н8в	02	02	C10	C10	Н9А	C10	C10	.C10	H11A	H11A	H11B	C10	C10	C10												Sus
												•							·							·	·			
		()	<u>.</u>	. (1	<u></u>	· •	(2	(6	. (2	. (,	3)	(/	. (9	3)	()	(2	2)	. ()	2)	3)	. (2	. (6	6	2}	()	(/	5)	. (9	(,
		110.7(5)	110.5(4)	106.4(4)	115.1(4)	120.4(4)	116.5(5)	123.1(5)	120.6(6)	120.2(7)	120.3(8)	119.9(7)	122.4(6)	119.3(13)	119.1(16)	121 (2)	121 (2)	120.3(16)	120.2(15)	120.2(13)	120.1(12)	119.3(10)	119.4(10)	108.1(5)	108.2(6)	108.2(7)	110(2)	109.5(16)	109.5(17)	
		-C12	-C10	-C14	-C14	-C15	-C19 ·	-C19	-C16	-C17	-C18	-C19	-C18	-H1	-H1	-H2	-H2	-н3	-Н3	-H4	-H4	-H5	-H5	-H7	-H7	-H7	-н8А	-H8B	н8С	
		-C10	-c13	-C13	-C13	-C14	-C14	-C14	-C15	-C16	-C17	-C18	-C19	-C1	-C1	-C2	-C2	-C3	-C3	-C4	-C4	-C5 .	-C5	٠-د٢	-C7	-67	-C8	-C8	. C8	
		C11	03	03	C10	C13	C15	C13	C14 -	C15	C16	C17	C14	C2	92	CJ	C3	C2	C4	C3	. 52	C4	90	N1	90	C8	C2	C.7	C2	
- 7 -	(Degrees)	113.5(2)	114.8(2)	113.2(2)	101.88(18)	106.2(2)	106.3(2)	114.2(4)	118.2(3)	126.1(3)	112(4)	122(4)	121.6(11)	119.0(11)	119.5(15)	119.6(10)	121.3(8)	120.2(8)	117.4(9)	122.3(5)	109.0(5)	111.7(4)	111.5(5)	113.2(4)	106.2(5)	109.6(5)	112.4(4)	109.8(5)	107.9(4)	
						•					Æ	Ą		~	_	. 10	9	, ,	2	7	o o	80	9	10	1.2	11	13	13	13	
	Bond Angles for: S886B	-02	-03	-N1	-03	-N1	-N1	60-	-C13	-C7	-H1A	-H1A	92-	-63	-C4	-C5	90-	-C7	-C5	-C7	-C8	-C8	90-	-C10	-C12	-C11	-c13	-C13	-C13	

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0 - 0 0 0 1			~ ~ ~ ~ ~ ~ ~ ~ ~ ~ .	,	

8 ; -13: 13 ; -25: 25

.. 8-

3040,

1.6, 27.5

0.348(1)

MoKa (graphite monochromator) 0.71073

0.0590, 0.0228 for 2941 Fo > 4sigma(Fo), 1.059

Refinement

Tot., Uniq. Data, R(int)

Refined mosaicity (deg)

Dataset

Radiation [Angstrom] Theta Min-Max [Deg]

Temperature (K)

206, sigma**2(F**2)+(0.0323*P)**2+0.21P

0.001, 0.000

-0.32, 0.18

Min. and Max. resd. dens. [e/Ang^3]

Max. and Av. Shift/Error

Npar, w**(-1) WR2, R1, S

S2106B : Aug 12 09:18:24 1999	Table S1 - Crystal Data a for: S2106B	Table S1 - Crystal Data and Details of the Structure Determination for: S2106B
		Crystal Data
NTARY MATERIA	Empirical Formula	C11 H13 C12 O3 P
	Formula Weight	295.08
	Crystal System	Orthorhombic
The Formation of Pyrophosphates; An NWR Study on the Mechanism	Space group	P212121 (No. 19)
and Stereochemistry of Pyrophosphate Formation from Chiral Dioxaphosphorinanes	a, b, c [Angstrom]	6.4383(10) 10.498(2) 19.571(3)
	V [Ang**3]	1322.8(4)
λα		7
Ron Hulst, Johanna M. Visser, N. Koen de Vries,	D(calc) [g/cm**3]	1.482
Robert W.J. Zijlstra, Huub Kooijman, Wilbertn J.J. Smeets, Anthony L. Spek and Ben L. Feringa	F(000)	809
	Mu(MoKa) [/mm]	9.0
COMPOUND 27	Crystal Size [mm]	0.15 x 0.15 x 0.25
1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	30.	Data Collection

7

Table S1 - Crystal Data and Details of the Structure Determination for: \$2106B	
Structure	
the	
of	
Details	
and	
l Data S2106B	
Crystal for: S2	
<u> </u>	
S1	
Table	

Contents

	olacement
	Dis
	Isotropic
	Equivalent Hydrogen at
	and non-
for: \$2106B	Table S2 - Final Coordinates and Equivalent Isotropic Displacement Parameters of the non-Hydrogen atoms for: S2106B
for:	- Final Parame for:
	22
	Table

Displacement		
Isotropic		
and		
- Hydrogen Atom Positions and Isotropic D		
Atom	ຸ	106B
gen	eter	S2106B
Hydro	Parameters	for:
Table S3		

(Angstrom)	
Distances	100
Bond	,
-1	
S 5	
Table	

Table S4 - (An)isotropic Displacement Parameters

(Degrees	
Angles	031105
Bond	402
1	

(Degrees)	
Angles	S2106B
Bond	for:
I	
98	
Table	

for: \$2106B

. Displacement	U(iso) [Ang^2]	0.014(4)	0.023(4)	0.034(5)	0.041(5)	0.036(5)	0.047(6)	0.054(6)	0.036(5)	0.031(4)	0.037(5)	0.036(5)	0.024(4)	0.034(5)
Hydrogen Atom Positions and Isotropic Parameters for: S2106B	z U	0.8707(7)	0.9811(9)	0.9961(8)	0.7829(10)	0.6637(10)	0.5955(11)	0.6454(11)	0.8312(10)	0.8909(8)	(6)6806.0	0,8238(9)	0.8843(8)	0.8965(9)
com Positions SB	≻₁	1.0376(15)	0.9480(17)	0.8029(18)	0.8764(19)	0.8428(19)	0.911(2)	1.016(2)	0.7091(18)	0.7568(18)	0.6637(19)	0.8025(18)	0.8931(16)	0.7465(19)
- Hydrogen Ato Parameters for: S2106B	×	0.225(2)	0.321(3)	0.263(3)	-0.163(3)	-0.175(3)	0.108(3)	0.394(4)	0.096(3)	-0.076(3)	0.126(3)	0.450(3)	0.536(3)	0.495(3)
Table S3	Atom	H3	HSA	н5В	H10	H11 '	H12	H13	H16A	H16B	H16C	H17A	H17B	H17C

ĵ

U(eq) [Ang^2]

N

×

Atom

C115

Final Coordinates and Equivalent Isotropic Displacement

Parameters of the non-Hydrogen atoms for: \$2106B

Table S2

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

0.0402(5) 0.0293(4) 0.0255(4) 0.0424(6) 0.0297(4) 0.0390(5) 0.0239(4) 0.0204(1) 0.0219(3) 0.0237(3) 0.0301(3) 0.0194(3) 0.0197(4)0.0230(4) 0.0296(1) 0.0414(1) 0.87307(7) 0.64040(8) 0.66858(8) 0.73929(7) 0.88070(8) 0.75146(7) 0.68108(8) 0.78178(7) 0.98672(2) 0.96298(2) 0.88419(5) 0.99864(4) 0.98338(6) 0.85862(7) 0.89472(6) 0.97171(7) 0.77252(2) 0.0824(3) 0.73241(14) 0.84918(13) 0.87695(14) 0.96049(14) 0.89939(15) 0.88149(17) 0.92444 (18) 0.98692(18) 0.2802(3) 1.00619(16) 0.4426(2) 0.82142(15) 0.97077(14) 1.17884(3) 1.09130(4) 1.02163(3) 1.02975(11) 1.00339(9) 0.91077(9) 0.2735(3) 0.1243(2) -0.0439(3) -0.0539(3) 0.1069(4) 0.2181(2) 0.2257(2) 0.31824(16) 0.1407(2) 0.06812(15) 0.01990(16) 0.05830(6) 0.49021(7) -0.10188(5)

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

C16

C17

C14

C12 C13

C10C11

65

	1					
	Table	SS - Bond for:	Distances (Ang S2106B	(Angstrom)		
	C17	-P1	2.0008(6)	C12	-c13	1.373(3)
	C115	-C14	1.7463(19)	C13	-C14	1.399(2)
•	P1	-02	1.5689(11)	ន	-H3	0.918(15)
	Pl	90-	1.5673(10)	C5	~H5A	0.983(18)
	P1 ·	-08	1.4516(11)	C5	-H5B	0.943(18)
	05	-C3	1.4749(16)	C10	-H10	1.01(2)
	90	-C5	1.4695(17)	C11	-H11	0.94(2)
	C3	C4	1.542(2)	C12	-H12	0.89(2)
	ເວ	6D-	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)
	60	-C10	1.391(2)	C17	-H17A	0.986(18)
	6 ,	-C14	1.389(2)	C17	-H17B	0.988(18)
	C10	-C11	1.392(2)	C17	-H17C	0.971(19)
	C11	-C12	1.382(3)			

Displacement Parameters

S4 - (An)isotropic

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

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

© 2000	Ame	rica	an (Che	mi	cal	So	cie	ty,	J . <i>A</i>	\ m	. C	hen	n. S	Soc	., H	luls	st ja	ı99	277	70k	Su	ppe	orti	ng	Inf	o P	'age	12
	60	C115	C115	C12	C11	C10	69	C10	C3	C3	90	C16	C5	C5	C3	СЗ	C3	C4	02	02	Гď	19	90	02	02	C17	C17	C17	Table
	-C14	-C14	-C14	-C13	-C12	-C11	-C10	-C9	-C9	-C9	-C5	-C4	-C4	-C4	-C4	-C4	-C4	-C3	-C3	-C3	- 06	-02	-P1	- P1	~P1	-P1	- P1	-P1	S6 - Bo fo
	-C13	-C13	-09	-C14	-C13	-C12	-C11	-C14	-C14	-C10	-C4	-C17	-C17	-C16	-C17	-C16	-C5	-C9	-C9	-C4	-C5	-C3	-08	-08	-06	-08	-06	-02	Bond Angles for: S2106B
	121.33(16)	117.79(14)	120.88(11)	119.37(17)	120.65(15)	119.45(17)	121.35(16)	117.80(13)	121.37(12)	120.77(12)	111.66(10)	109.65(12)	106.08(10)	110.26(11)	109.59(11)	113.36(11)	107.63(11)	114.80(12)	106.89(10)	109.35(10)	118.08(8)	119.22(8)	113.65(6)	114.23(6)	106.13(5)	112.48(5)	104.55(4)	104.92(4)	(Degrees)
·	Н17В	H17A	H17A	C4	C4	C4	H16B	H16A	H16A	C4	C4	C4	C14	C1'2	C13	C11	C12	C10	C11	C9 ,	н5а	C4	C4	90	90	С9	C4	02	
	-C17	-C17	-C17	-C17	-C17	-C17	-C16	-C16	-C16	-C16	-C16	-C16	-C13	-C13	-C12	-C12	-C11	-C11	-C10	-C10	-c5	-05	-C5	-03	-C5	-03	-c3	-C3	
,	-н17С	-H17C	-H17B	-H17C	-н17в	-H17A	-H16C	-H16C	-H16B	-H16C	-н16в	-H16A	~H13	-H13	-H12	-H12	-H11	-H11	-H10	-H10	-H5B	-H5B	-H5A	-H5B	-H5A	-H3	-Н3	-Н3	
	107.5(15)	106.4(15)	110.0(14)	110.5(11)	111.5(11)	110.7(11)	110.8(15)	110.5(16)	108.8(14)	109.6(12)	108.6(10)	108.6(11)	113.6(14)	126.9(13)	117.8(13)	121.5(13)	123.5(12)	117.0(12)	122.3(11)	116.1(11)	111.8(16)	110.4(10)	110.3(10)	104.3(11)	108.2(11)	110.6(9)	108.9(9)	105.9(9)	

Sub 12.