Supporting Information for

Synthesis of Phosphole-2,5- Dicarboxylic Acids via a [1,5] Shift of Carbon Dioxide around the phosphole nucleus.

Mohand Melaimi, Louis Ricard, François Mathey

and Pascal Le Floch*

Laboratoire "Hétéroéléments et Coordination", UMR CNRS 7653, Ecole Polytechnique, 91128 Palaiseau Cedex, France.

General :

All reactions were routinely performed under an inert atmosphere of argon or nitrogen by using Schlenk and glove-box techniques and dry deoxygenated solvents. Dry THF and hexanes were obtained by distillation from Na/benzophenone and dry CH_2Cl_2 and $CDCl_3$ from P_2O_5. Dry CD_2Cl_2 was distilled and stored, like $CDCl_3$, on 4Å Linde molecular sieves. Nuclear magnetic resonance spectra were recorded on a Bruker Avance 300 spectrometer operating at 300 MHz for ^{1}H, 75.5 MHz for ^{13}C and 121.5 MHz for ^{31}P. Solvent peaks are used as internal reference relative to Me_4Si for ^{1}H and ^{13}C chemical shifts (ppm); ^{31}P chemical shifts are relative to a 85% H_3PO_4 external reference and coupling constants are expressed in Hertz. The following abbreviations are used: b; broad, singlet; d, doublet; t, triplet; m, multiplet; q, quadruplet. Mass spectra were obtained at 70 eV with a HP 5989B spectrometer coupled to a HP 5980 chromatograph by the direct inlet method. Elemental analyses were performed by the "Service d'analyse du CNRS", at Gif sur Yvette, France.

Synthesis of Phospholes-2,5-Dicarboxylic Acid. A solution of *n*-BuLi (25 mL, 1.6M in hexanes, 40 mmol, 2 equiv.) was added at –80°C to a solution of zirconocene dichloride (5.84 g, 20 mmol) in THF (200 mL). The resulting solution was then stirred for1 hour at –80°C and 1-trimethylsilylpropyne (4.49 g, 40 mmol) was added. The resulting mixture was then allowed to warm-up to room temperature and stirred for an additional 17 hours. After evaporation of the THF, dichloromethane was added (2 x 75 ml) and the resulting orange solution was filtrated. After evaporation of the solvent, zirconacyclopentadiene was obtained as a red-brownish powder. Dichloromethane was added (100 mL) and the metallacycle was reacted with PCl_3 (7.5 g, 17 mmol) at 40 °C during 2 hour. After cooling the resulting solution to room temperature, the solvent was evaporated and freshly distilled hexane was added (3 x 50 ml). After filtration and evaporation of hexane, the 1-P-chloro-2,5-bis(trimethylsilyl)-3,4-dimethylphosphole was obtained as a very moisture sensitive yellow oil. After THF (100 mL) was added, the freshly prepared chlorophosphole was reacted with excess lithium (3.28 g, 47 mmol) at room temperature. The reaction was followed by ^{31}P NMR. After 45 minutes, all the starting material was converted into the 1,1'-biphosphole P-P dimer. After an additional 1 hour, a ^{31}P NMR control indicated the complete formation of anion **1**. The excess lithium was then eliminated by filtration. Carbon dioxide was bubbled into the solution of 2,5-bis(trimethylsilyl) lithium phospholide **1** (6.0 mmol) in THF (50 mL) at room temperature for 2 minutes. After checking the formation of anion **5** by ^{31}P NMR, the electrophile (6.0 mmol or 3.0 mmol for the synthesis of **10** and **11**) was added. After 30 minutes stirring, the reaction was completed and LiI (100 mmol) was added. After 12 hours stirring at room temperature,

the solvent was evaporated, methanol (40 mL) and then TMSCL (18.0 mmol) were added. The resulting solution was stirred for 5 minutes and celite (2 g)was added. After evaporation of the solvent the coated silicagel was dropped onto the top of a silicagel packed column for chromatography and the phosphole was eluted with methanol as eluent. After evaporation of the methanol, the product was obtained as a pale yellow powder.

3,4-dimethyl-5-trimethylsilanyl –(O-trimethylsilanyl) 2-carboxylic acid-phospholide (**4**)

^{1}H NMR (THF d_8) : δ 0.26 (d, $^4J_{PH}$ = 1.0 Hz, 9H, P-C-Si-CH_3), 0.38 (d, $^4J_{PH}$ = 1.0 Hz, 9H, P-C-Si-CH_3), 2.05 (s, 3H, TMS-C=C-CH_3), 2.20 (s, 3H, COO-C=C-CH_3); ^{13}C NMR (THF d_8) : δ 0.0 (P-C-Si-C*H*3), 1.4 (COO-Si*C*H$_3$), 15.6 (TMS-C=C-*C*H3), 17.2 (COO-C=C-*C*H3), 131.0 (d, $^2J_{PC}$ = 28 Hz, 23.9 Hz, P-C_5), 134.5 (d, $^2J_{PC}$ = 8.6 Hz, P-C=C_6), 135.1 (d, $^2J_{PC}$ = 7.2 Hz, P-C=C_3), 134.5 (d, $^2J_{PC}$ = 8.6 Hz, P-C=C_3), 148.9 (d, $^2J_{PC}$ = 52.3 Hz, P-C_2), 172.2 (d, $^2J_{PC}$ = 27.4 Hz, P-C-*C*OOTMS), ^{31}P NMR (THFd_8) : δ 160.0; MS (NCI) : m/z (% relative intensitiy): 299 (100); Anion **4** was found to be too oxygen and moisture sensitive for the recording of elemental data.

3,4-dimethyl-2,5-bis-(O-trimethylsilanyl) carboxylic acid-phospholide (**5**)

^{1}H NMR (THFd_8): δ 0.39 (s, 18 H, COOSi-CH_3), 2.3 (d, 6 H, P-C=C-CH_3); ^{13}C NMR (THFd_8) : δ -0.4 (Si*C*H$_3$), 15.0 (P-C=C-CH*3*), 133.9 (P-C=*C*), 137.4 (d, $^2J_{PC}$ = 28 Hz, P-*C*=C), 171.1 (d, $^2J_{PC}$ = 28 Hz, P-C-*C*OOTMS); ^{31}P NMR (THFd_8) : δ 165.0; MS (NCI) : m/z (% relative intensitiy): 343 (100). Anion **5** was found to be too oxygen and moisture sensitive for the recording of elemental analysis.

1-benzyl-3',4'-dimethyl-1'H-phosphole-2',5'-dicarboxylic acid (**6**)

^{1}H NMR (CD$_3$OD) : δ 2.15 (d, $^4J_{PH}$ = 4.3 Hz, 6 H, P-C=C-CH_3), 3.45 (d, $^2J_{PH}$ = 4.6 Hz, 2 H, P-CH_2), 6.90-7.03 (m, 5 H, H$_{ARO}$); ^{13}C NMR (CD$_3$OD) : δ 15.6 (P-C=C-*C*H$_3$), 32.3 (d, $^1J_{PC}$ =26.7 Hz, P-*C*H$_2$), 126.1 (C$_{ARO}$-p), 128.2 (C$_{ARO}$-m), 128.8 (C$_{ARO}$-o), 138.3 (d, $^2J_{PC}$ =6.4 Hz, P-C=*C*), 142.2 (C$_{ARO}$-ipso), 154.9 (d, $^2J_{PC}$ =10.9 Hz, P-*C*=C), 172.4 (d, $^3J_{PC}$ =18.6 Hz, *C*OOD); ^{31}P NMR (CD$_3$OD) : δ 15.2; MS (PCI) : m/z (% relative intensitiy): 291 (M+1, 100). Anal. Calcd. For $C_{15}H_{15}O_4P$: C, 62.07; H, 5.21. Found : C, 61.82; H, 5.42.

2-(3',4'-dimethyl-1'H-phosphole-2',5'-dicarboxylic acid) ethylacetate (**7**)

^{1}H NMR (CD$_3$OD): δ 1.09 (t, $^3J_{HH}$ = 7.1 Hz, 3H, O-CH$_2$-CH_3), 2.38 (d, $^4J_{PH}$ = 4.6 Hz, 6 H, P-C=C-CH_3), 3.11 (d, $^3J_{PH}$ = 3.3 Hz, 6 H, P-CH$_2$-CH_2), 3.88 (q, $^3J_{HH}$ = 7.1 Hz, 2H, O-CH_2); ^{13}C NMR (CD$_3$OD): δ 14.6 (O-CH$_2$- *C*H$_3$), 15.8 (p-C=C-*C*H$_3$), 19.8 (P-*C*H$_2$), 30.9 (d, $^2J_{PC}$ =34 Hz, P- *C*H$_2$), 61.6 O-*C*H$_2$), 142.3 (P-C=*C*), 155.6.(d, $^2J_{PC}$ =11.4 Hz, P-*C*=C), 171.3 (d, $^2J_{PC}$ =5 Hz, *C*OOD), 171.6 (d, $^3J_{PC}$ =19.4 Hz, *C*OOEt); ^{31}P NMR (CD$_3$OD): δ -1.0; MS (PCI) : m/z (% relative intensitiy): 287 (M+1, 100); Anal. Calcd. For $C_{12}H_{15}O_6P$: C, 50.36; H, 5.28. Found : C, 50.79; H, 5.52.

3-(3',4'-dimethyl-1'H-phosphole-2',5'-dicarboxylic acid) ethylpropionate (**8**)

^{1}H NMR (CD_3OD): δ 1.02 (t, $^3J_{HH}$ = 7.2 Hz, 3H, O-CH_2-CH_3), 1.88 (q, $^2J_{PH}$ = $^3J_{HH}$ = 7.3 Hz, 2 H, P-CH_2), 2.26 (m, 2H, P-CH_2-CH_2), 2.28 (d, $^4J_{PH}$ = 5.1 Hz, 6 H, P-C=C-CH_3), 3.86 (q, $^3J_{HH}$ = 7.2 Hz, 2H, O-CH_2); ^{13}C NMR (CD_3OD) : δ 14.4 (O-CH_2- CH_3), 15.9 (p-C=C-CH_3), 19.8 (d, $^1J_{PC}$ =25.5 Hz, P-CH_2), 31.2 (P-CH_2-CH_2), 61.9 (O-CH_2), 138.1 (P-C=C), 159.0 (d, $^2J_{PC}$ =7.3 Hz, P-C=C), 168.2 (d, $^3J_{PC}$ =19.7 Hz, COOEt), 174.4 (d, $^2J_{PC}$ =4.7 Hz, COOD); ^{31}P NMR (CD_3OD) : δ 14.9. Anal. MS (PCI) : m/z (% relative intensitiy): 301 (M+1, 100); Anal. Calcd. For $C_{13}H_{17}O_6P$: C, 52.0; H, 5.71. Found : C, 51.82; H, 6.22.

3-(3',4'-dimethyl-1'H-phosphole-2',5'-dicarboxylic acid) propionitrile (**9**)

^{1}H NMR (THF-d_8) : δ 2.01-2.09 (m, 2 H, P-CH_2), 2.37 (d, $^4J_{PH}$ = 5.1 Hz, 2 H, P-CH_2), 2.38-2.45 (m, 2H, P-CH_2-CH_2), 11.25 (br s, 1 H, COOH); ^{13}C NMR (THF-d_8) : δ 11.3 (P-CH_2-CH_2), 13.2 (p-C=C-CH_3), 18.6 (d, $^1J_{PC}$ =29.4 Hz, P-CH_2), 117.2 (d, $^3J_{PC}$ = 6.7 Hz, CN), 134.4 (d, $^2J_{PC}$ = 3.0 Hz, P-C=C), 157.1 (d, $^1J_{PC}$ = 10.6 Hz, P-C=C), 163.7 (d, $^2J_{PC}$ = 19.6 Hz, COOH); ^{31}P NMR (THF-d_8) : δ 11.6. MS (PCI) : m/z (% relative intensitiy): 301 (M+1, 100).Anal. Calcd. For $C_{11}H_{12}NO_4P$: C, 52.18; H, 4.78. Found : C, 51.87; H, 5.11.

α,α' -bis (3',4'-dimethyl-1'H-phosphole-2',5'-dicarboxylic acid) -o-xylene (**10**)

^{1}H NMR (CD_3OD) : δ 2.24 (d, $^4J_{PH}$ = 5.0 Hz, 12 H, P-C=C-CH_3), 3.34 (d, $^2J_{PH}$ = 5.2 Hz, 4 H, P-CH_2), 6.67 (br dd, $^3J_{HH}$ = 8.6 Hz, $^4J_{HH}$ = 5.2 Hz, 2 H, H-o), 6.79 (br dd, $^3J_{HH}$ = 8.6 Hz, $^4J_{HH}$ = 5.2 Hz, 2 H, H-o-p), 6.76 (t, $^3J_{PH}$ = 7.6 Hz, 1 H, H-m-m); ^{13}C NMR (CD_3OD) : δ 15.6 (P-C=C-CH_3), 29.5 (d, $^1J_{PC}$ =29.0 Hz, P-CH_2), 125.8 (C_{ARO}-o), 129.8 (C_{ARO}-m), 135.0 (d, 5.8 Hz, C_{ARO}-ipso), 138.2 (d, $^3J_{PC}$ =3.5 Hz, P-C=C), 159.4.(d, $^2J_{PC}$ =9.2 Hz, P-C=C), 168.5 (d, $^3J_{PC}$ =19.5 Hz, COOD); ^{31}P NMR (CD_3OD) : δ 19.4; MS (PCI) : m/z (% relative intensitiy): 503 (M+1, 100); Anal. Calcd. For $C_{24}H_{24}O_8P_2$: C, 57.38; H, 4.82. Found : C, 56.89; H, 5.53.

α,α' -bis (3',4'-dimethyl-1'H-phosphole-2',5'-dicarboxylic acid) -m-xylene (**11**)

^{1}H NMR (CD_3OD) : δ 2.19 (d, $^4J_{PH}$ = 5.1 Hz, 12 H, P-C=C-CH_3), 3.35 (d, $^2J_{PH}$ = 5.5 Hz, 4 H, P-CH_2), 6.37 (br s, 1 H, H-o-o), 6.45 (br d, 2 H, H-o-p), 6.76 (t, $^3J_{PH}$ = 7.6 Hz, 1 H, H-m-m); ^{13}C NMR (CD_3OD) : δ 14.3 (P-C=C-CH_3), 31.7 (d, $^1J_{PC}$ =27.2 Hz, P-CH_2), 125.0 (C_{ARO}-o-p), 126.4 and 127.4 (C_{ARO}-o-o and C_{ARO}-m-m), 135.8 (d, $^2J_{PC}$ =6.1 Hz, C_{ARO}-ipso), 137.5 (d, $^3J_{PC}$ =1.7 Hz P-C=C), 158.7.(d, $^2J_{PC}$ =9.4 Hz, P-C=C), 167.5 (d, $^3J_{PC}$ =20.0 Hz, COOD); ^{31}P NMR (CD_3OD) : δ 20.1; MS (PCI) : m/z (% relative intensitiy): 503 (M+1, 100); Anal. Calcd. For $C_{24}H_{24}O_8P_2$: C, 57.38; H, 4.82. Found : C, 56.62; H, 5.46.

TABLE 1: X-RAY-DATA FOR COMPOUND 10

Compound	**10**
Molecular formula	C26H32O10P2
Molecular weight	566.46
Crystal habit	pale yellow plate
Crystal dimensions(mm)	0.20x0.20x0.08
Crystal system	'Triclinic'
Space group	'P-1
a(Å)	8.9958(3)
b(Å)	12.7612(3)
c(Å)	13.2746(5)
a(°)	68.4460(10)
b(°)	80.8930(10)
g(°)	71.0940(10)
V(Å3)	1339.53(7)
Z	2
d(g-cm-3)	1.404
F000	596
m(cm-1)	0.219
Absorbtion corrections	? ; 0.9576 min, 0.9827 max
Difractometer	KappaCCD
X-ray source	MoKa
l(Å)	0.71069
Monochromator	graphite
T (K)	150.0(10)
Scan mode	'phi
Maximum q	27.48
HKL ranges	-11 11 ; -16 16 ; -17 13
Reflections measured	9565
Independant reflections	6122
Rint	0.0291
Reflections used	4549
Criterion	>2sigma(I)
Refinement type	Fsqd
Hydrogen atoms	mixed
Parameters refined	355
Reflections / parameter	12
wR2	0.1300
R1	0.0456
Flack's parameter	not applicable
Weighs a, b1	0.0680 ; 0.1939
GoF	1.034
difference peak / hole (e Å-3)	0.488(0.059) / -0.325(0.059)

TABLE 2: Atomic Coordinates (A x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for compound 10.

atom	x	y	z	U(eq)
P(1)	1312(1)	4397(1)	7998(1)	24(1)
P(2)	4892(1)	767(1)	6454(1)	24(1)
O(1)	1870(2)	6723(1)	7171(1)	36(1)
O(2)	3207(2)	6588(1)	8514(1)	41(1)
O(3)	826(2)	1309(1)	10094(1)	40(1)
O(4)	293(2)	2315(1)	8362(1)	29(1)
O(5)	4788(2)	4055(1)	4530(1)	38(1)
O(6)	6670(2)	2578(1)	5511(1)	36(1)
O(7)	2214(2)	-1162(1)	6339(1)	36(1)
O(8)	3881(2)	-1379(1)	7534(1)	36(1)
C(1)	2101(2)	5051(1)	8706(2)	25(1)
C(2)	2220(2)	4430(2)	9785(2)	25(1)
C(3)	1741(2)	3352(2)	10099(2)	26(1)
C(4)	1319(2)	3183(1)	9246(2)	24(1)
C(5)	3100(2)	3800(2)	7205(2)	26(1)
C(6)	4422(2)	2901(2)	7894(2)	23(1)
C(7)	5340(2)	3286(2)	8361(2)	30(1)
C(8)	6445(2)	2506(2)	9113(2)	33(1)
C(9)	6677(2)	1310(2)	9401(2)	33(1)
C(10)	5841(2)	911(2)	8905(2)	28(1)
C(11)	4707(2)	1681(2)	8158(2)	23(1)
C(12)	3848(2)	1161(2)	7662(2)	25(1)
C(13)	4416(2)	2140(2)	5337(2)	25(1)
C(14)	3350(2)	2185(2)	4693(2)	26(1)
C(15)	2731(2)	1160(2)	5111(2)	25(1)
C(16)	3396(2)	361(2)	6049(2)	26(1)
C(17)	2451(2)	6186(2)	8141(2)	27(1)
C(18)	2770(3)	4743(2)	10615(2)	34(1)
C(19)	1770(3)	2569(2)	11257(2)	37(1)
C(20)	788(2)	2181(2)	9301(2)	24(1)
C(21)	5287(2)	3023(2)	5080(2)	27(1)
C(22)	2860(3)	3100(2)	3616(2)	37(1)
C(23)	1502(2)	1099(2)	4508(2)	32(1)
C(24)	3085(2)	-782(2)	6639(2)	27(1)
O(10)	160(2)	396(1)	8178(1)	34(1)
O(9)	7945(2)	4239(1)	5249(1)	41(1)
C(26)	9248(3)	642(2)	7286(2)	40(1)
C(25)	-996(3)	3912(2)	6068(3)	60(1)

U(eq) is defined as 1/3 the trace of the Uij tensor.

TABLE 3: Bond lengths (A) and angles (deg) for compound 10.

P(1)-C(1)	1.803(2)	P(1)-C(4)	1.804(2)
P(1)-C(5)	1.880(2)	P(2)-C(16)	1.804(2)
P(2)-C(13)	1.805(2)	P(2)-C(12)	1.875(2)
O(1)-C(17)	1.316(2)	O(2)-C(17)	1.221(2)
O(3)-C(20)	1.213(2)	O(4)-C(20)	1.323(2)
O(5)-C(21)	1.218(2)	O(6)-C(21)	1.314(2)
O(7)-C(24)	1.224(2)	O(8)-C(24)	1.324(2)
C(1)-C(2)	1.360(3)	C(1)-C(17)	1.477(2)
C(2)-C(3)	1.468(2)	C(2)-C(18)	1.497(3)
C(3)-C(4)	1.356(3)	C(3)-C(19)	1.494(3)
C(4)-C(20)	1.479(2)	C(5)-C(6)	1.502(3)
C(6)-C(7)	1.398(3)	C(6)-C(11)	1.409(2)
C(7)-C(8)	1.385(3)	C(8)-C(9)	1.382(3)
C(9)-C(10)	1.382(3)	C(10)-C(11)	1.395(3)
C(11)-C(12)	1.512(2)	C(13)-C(14)	1.357(3)
C(13)-C(21)	1.485(2)	C(14)-C(15)	1.473(2)
C(14)-C(22)	1.496(3)	C(15)-C(16)	1.362(3)
C(15)-C(23)	1.501(3)	C(16)-C(24)	1.474(2)
O(10)-C(26)#1	1.429(3)	O(9)-C(25)#1	1.415(3)
C(26)-O(10)#1	1.429(3)	C(25)-O(9)#1	1.415(3)

C(1)-P(1)-C(4)	88.70(8)	C(1)-P(1)-C(5)	101.7(1)
C(4)-P(1)-C(5)	103.74(8)	C(16)-P(2)-C(13)	88.89(8)
C(16)-P(2)-C(12)	99.9(1)	C(13)-P(2)-C(12)	104.12(8)
C(2)-C(1)-C(17)	126.0(2)	C(2)-C(1)-P(1)	112.8(1)
C(17)-C(1)-P(1)	121.1(2)	C(1)-C(2)-C(3)	112.6(2)
C(1)-C(2)-C(18)	126.8(2)	C(3)-C(2)-C(18)	120.6(2)
C(4)-C(3)-C(2)	113.0(2)	C(4)-C(3)-C(19)	126.9(2)
C(2)-C(3)-C(19)	120.1(2)	C(3)-C(4)-C(20)	125.8(2)
C(3)-C(4)-P(1)	112.6(1)	C(20)-C(4)-P(1)	121.5(1)
C(6)-C(5)-P(1)	114.1(1)	C(7)-C(6)-C(11)	118.3(2)
C(7)-C(6)-C(5)	118.7(2)	C(11)-C(6)-C(5)	122.8(2)
C(8)-C(7)-C(6)	122.0(2)	C(9)-C(8)-C(7)	119.5(2)
C(8)-C(9)-C(10)	119.4(2)	C(9)-C(10)-C(11)	122.0(2)
C(10)-C(11)-C(6)	118.7(2)	C(10)-C(11)-C(12)	118.2(2)
C(6)-C(11)-C(12)	123.1(2)	C(11)-C(12)-P(2)	115.0(1)
C(14)-C(13)-C(21)	124.6(2)	C(14)-C(13)-P(2)	112.3(1)
C(21)-C(13)-P(2)	122.5(1)	C(13)-C(14)-C(15)	113.3(2)
C(13)-C(14)-C(22)	127.0(2)	C(15)-C(14)-C(22)	119.5(2)
C(16)-C(15)-C(14)	112.2(2)	C(16)-C(15)-C(23)	128.0(2)
C(14)-C(15)-C(23)	119.8(2)	C(15)-C(16)-C(24)	125.4(2)
C(15)-C(16)-P(2)	112.8(1)	C(24)-C(16)-P(2)	121.5(1)
O(2)-C(17)-O(1)	123.0(2)	O(2)-C(17)-C(1)	124.5(2)
O(1)-C(17)-C(1)	112.5(2)	O(3)-C(20)-O(4)	122.4(2)
O(3)-C(20)-C(4)	125.3(2)	O(4)-C(20)-C(4)	112.2(2)
O(5)-C(21)-O(6)	122.7(2)	O(5)-C(21)-C(13)	124.3(2)
O(6)-C(21)-C(13)	113.0(2)	O(7)-C(24)-O(8)	122.8(2)
O(7)-C(24)-C(16)	124.6(2)	O(8)-C(24)-C(16)	112.7(2)

TABLE 4: Anisotropic displacement parameters (A^2 x 10^3) for compound 10.

atom	U11	U22	U33	U23	U13	U12
P(1)	29(1)	19(1)	25(1)	-5(1)	-7(1)	-8(1)
P(2)	29(1)	22(1)	24(1)	-9(1)	-4(1)	-9(1)
O(1)	57(1)	26(1)	31(1)	-3(1)	-12(1)	-22(1)
O(2)	56(1)	30(1)	44(1)	-1(1)	-24(1)	-21(1)
O(3)	65(1)	28(1)	31(1)	-1(1)	-14(1)	-24(1)
O(4)	37(1)	26(1)	30(1)	-7(1)	-8(1)	-16(1)
O(5)	46(1)	29(1)	40(1)	-4(1)	-12(1)	-18(1)
O(6)	39(1)	35(1)	40(1)	-6(1)	-12(1)	-20(1)
O(7)	50(1)	28(1)	39(1)	-6(1)	-16(1)	-20(1)
O(8)	48(1)	24(1)	39(1)	-3(1)	-18(1)	-16(1)
C(1)	26(1)	21(1)	30(1)	-10(1)	-4(1)	-6(1)
C(2)	24(1)	23(1)	29(1)	-11(1)	-5(1)	-3(1)
C(3)	26(1)	23(1)	26(1)	-7(1)	-4(1)	-4(1)
C(4)	24(1)	20(1)	26(1)	-7(1)	-5(1)	-5(1)
C(5)	34(1)	23(1)	24(1)	-6(1)	-3(1)	-13(1)
C(6)	26(1)	25(1)	20(1)	-10(1)	1(1)	-9(1)
C(7)	28(1)	31(1)	38(1)	-18(1)	1(1)	-11(1)
C(8)	26(1)	44(1)	41(1)	-26(1)	-5(1)	-9(1)
C(9)	28(1)	40(1)	30(1)	-15(1)	-6(1)	-4(1)
C(10)	30(1)	26(1)	26(1)	-10(1)	-1(1)	-6(1)
C(11)	25(1)	26(1)	20(1)	-9(1)	2(1)	-11(1)
C(12)	32(1)	24(1)	24(1)	-9(1)	0(1)	-13(1)
C(13)	30(1)	26(1)	23(1)	-9(1)	-1(1)	-11(1)
C(14)	31(1)	27(1)	24(1)	-10(1)	-1(1)	-12(1)
C(15)	26(1)	27(1)	26(1)	-12(1)	-2(1)	-9(1)
C(16)	31(1)	23(1)	28(1)	-13(1)	-3(1)	-9(1)
C(17)	30(1)	20(1)	32(1)	-9(1)	-7(1)	-6(1)
C(18)	43(1)	35(1)	30(1)	-14(1)	-4(1)	-15(1)
C(19)	56(1)	32(1)	26(1)	-6(1)	-7(1)	-18(1)
C(20)	24(1)	22(1)	26(1)	-6(1)	-4(1)	-7(1)
C(21)	35(1)	28(1)	22(1)	-9(1)	0(1)	-15(1)
C(22)	54(1)	35(1)	28(1)	-4(1)	-12(1)	-22(1)
C(23)	34(1)	34(1)	30(1)	-10(1)	-9(1)	-12(1)
C(24)	32(1)	21(1)	30(1)	-9(1)	-6(1)	-8(1)
O(10)	39(1)	31(1)	35(1)	-8(1)	-6(1)	-16(1)
O(9)	45(1)	39(1)	41(1)	-6(1)	-15(1)	-18(1)
C(26)	41(1)	53(1)	33(1)	-17(1)	-2(1)	-21(1)
C(25)	61(2)	52(1)	70(2)	-19(1)	-38(2)	-6(1)

The anisotropic displacement factor exponent takes the form
2 pi^2 [h^2a*^2U(11) +...+ 2hka*b*U(12)]

TABLE 5: Hydrogen Coordinates (A x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for compound 10.

atom	x	y	z	U(eq)
H(1)	2046	7380	6893	54
H(4)	200	1674	8380	44
H(6)	7081	3119	5391	55
H(8)	3702	-2034	7828	54
H(5A)	2790	3439	6757	32
H(5B)	3486	4463	6706	32
H(7)	5202	4106	8157	36
H(8A)	7038	2791	9429	40
H(9)	7404	768	9934	39
H(10)	6046	87	9079	34
H(12A)	2807	1733	7444	30
H(12B)	3662	444	8224	30
H(18A)	2892	5536	10288	51
H(18B)	3783	4178	10874	51
H(18C)	1995	4719	11224	51
H(19A)	1221	1987	11349	56
H(19B)	1244	3043	11719	56
H(19C)	2861	2162	11463	56
H(22A)	1725	3482	3651	56
H(22B)	3107	2727	3059	56
H(22C)	3426	3690	3433	56
H(23A)	1252	352	4875	48
H(23B)	1902	1150	3765	48
H(23C)	551	1754	4489	48
H(10A)	-82	-132	8717	50
H(9A)	7206	4834	5277	61
H(26A)	8180	1127	7389	60
H(26B)	9204	-98	7245	60
H(26C)	9735	1067	6610	60
H(25A)	-597	4573	5972	90
H(25B)	-115	3235	6019	90
H(25C)	-1542	3701	6781	90