Supporting Information for JA001722T

Two New Iodine-Capped Carbon Rods

Kunxiu Gao and Nancy S. Goroff
Dept. of Chemistry, SUNY-Stony Brook

Experimental procedures

3: To a solution of compound **7**, bis(trimethylsilyl)hexatriyne, (46 mg, 0.24 mmol) in 4.5 mL DMF was added $AgNO_3$ (4.3 mg, 0.03 mmol) and *N*-iodosuccinimide (116 mg, 0.5 mmol). The reaction vessel was wrapped in aluminum foil, and the mixture stirred for 3 h. Cold water was then added, precipitating the product. The solid was filtered and dissolved in ether. This solution was then washed with water and dried with $NaSO_4$. Removing solvent *in vacuo* afforded **3** as a yellow solid (41.3 mg, 65%): M.p. 100 °C *(explodes)*; ^{13}C NMR ($CDCl_3$) δ 78.5, 59.7, 0.9; ^{13}C NMR (DMSO-d_6) δ 76.3, 58.8, 14.6; MS (EI): *m/z* (%): 326 (100) [M^+ (C_6I_2), 199 (35) [M^+ – I], 127 (38) [I^+], 72 (25) [M^+ – 2(I)]; HRMS (EI) calcd for C_6I_2 [M^+] 325.8090, found 325.8086.

4: To a solution of compound **10**, bis(trimethylsilyl)octatetrayne, (49 mg, 0.22 mmol) in 5 mL DMF was added $AgNO_3$ (10 mg, 0.05 mmol) and *N*-iodosuccinimide (101 mg, 0.44 mmol). The reaction vessel was wrapped in aluminum foil, and the mixture stirred for 3 h. Cold water was then added, precipitating the product. The solid was filtered and then dissolved in ether. This solution was washed with water three times, then dried with $MgSO_4$. Removing solvent *in vacuo* afforded **4** as a yellow solid (50 mg, 71%): M.p. 85 °C *(explodes)*; ^{13}C NMR (DMSO-d_6) δ 77.4, 62.7, 58.3, 17.9.

#JACS manuscript number JA001722T-31-93
Crystallographic Data for Diiodotriacetyelene Triphenylphosphineoxide

Empirical Formula C24H15I2PO
Formula Weight 616.2
Crystal System monoclinic
Lattice Parameters a = 10.915(1) ≈
b = 14.051(1) ≈
c = 15.399(2) ≈
b = 97.84(1)∞
vol = 2339.8(4) ≈
Space Group P21/n (#14)
Z value 4
Dcalc 1.749 g/cm3
_ (Mo K_) 27.7 cm-1
Fobs (unique I > 3_) 1342
No. variables 253
R, Rw 0.057, 0.053

Positional parameters and B(eq) for Diiodotriacetylene triphenylphosphineoxide

atom	x	y	z	B(eq)
I(1)	1.02581(6)	0.54199(4)	0.19009(4)	9.36(2)
I(2)	0.74360(6)	-0.05040(5)	0.50183(4)	10.04(2)
P(1)	0.9256(2)	0.2175(2)	0.9355(2)	8.39(7)
O(1)	0.8841(5)	0.3137(4)	0.9050(4)	9.4(1)
C(1)	0.9630(9)	0.4315(7)	0.2466(7)	10.4(3)
C(2)	0.9277(9)	0.3597(8)	0.2794(6)	10.1(2)
C(3)	0.8880(9)	0.2779(7)	0.3195(7)	10.2(2)
C(4)	0.8616(9)	0.2093(8)	0.3597(7)	10.2(3)
C(5)	0.8285(8)	0.1299(8)	0.4027(7)	10.3(3)
C(6)	0.8016(9)	0.0597(8)	0.4404(7)	10.7(3)
C(7)	0.9509(8)	0.1401(6)	0.8461(6)	8.4(2)
C(8)	1.0306(8)	0.1704(6)	0.7947(6)	9.0(2)
C(9)	1.0560(10)	0.1185(9)	0.7248(8)	14.1(3)
C(10)	0.9994(8)	0.0424(7)	0.7004(6)	9.1(2)
C(11)	0.9121(9)	0.0029(6)	0.7541(7)	11.5(3)
C(12)	0.8845(8)	0.0573(7)	0.8267(7)	10.4(2)
C(13)	0.8130(8)	0.1631(6)	0.9958(6)	8.4(2)
C(14)	0.6909(10)	0.1733(6)	0.9623(6)	10.6(3)
C(15)	0.5947(10)	0.1347(7)	1.0021(7)	11.9(3)
C(16)	0.6302(12)	0.0851(8)	1.0823(9)	14.0(3)
C(17)	0.7481(11)	0.0789(7)	1.1195(7)	12.2(3)
C(18)	0.8422(8)	0.1202(6)	1.0750(6)	9.6(2)
C(19)	1.0689(8)	0.2178(6)	1.0069(6)	8.9(2)
C(20)	1.0888(9)	0.2942(7)	1.0634(7)	9.9(2)
C(21)	1.1977(11)	0.2915(7)	1.1270(8)	11.8(3)
C(22)	1.2823(10)	0.2225(8)	1.1217(7)	11.7(3)
C(23)	1.2621(10)	0.1491(8)	1.0668(8)	11.9(3)
C(24)	1.1571(9)	0.1464(6)	1.0077(6)	10.1(2)
H(1)	1.0744	0.2302	0.8099	10.660
H(2)	1.1157	0.1490	0.6876	16.039
H(3)	1.0228	0.0033	0.6500	10.972
H(4)	0.8704	-0.0591	0.7398	13.168
H(5)	0.8200	0.0364	0.8624	12.277
H(6)	0.6709	0.2095	0.9071	12.387
H(7)	0.5057	0.1420	0.9770	13.462
H(8)	0.5656	0.0507	1.1084	16.324
H(9)	0.7695	0.0467	1.1757	13.853
H(10)	0.9314	0.1176	1.1019	11.194
H(11)	1.0289	0.3495	1.0587	11.754
H(12)	1.2097	0.3382	1.1758	13.968

H(13)	1.3617	0.2267	1.1600	14.150
H(14)	1.3245	0.0966	1.0674	14.422
H(15)	1.1406	0.0922	0.9672	11.672

Intramolecular Distances

atom	atom	distance	atom	atom	distance
I1	C1	1.95(1)	C13	C14	1.37(1)
I2	C6	1.96(1)	C13	C18	1.36(1)
P1	O1	1.483(5)	C14	C15	1.40(1)
P1	C7	1.805(8)	C14	H6	0.989
P1	C13	1.808(8)	C15	C16	1.43(1)
P1	C19	1.785(8)	C15	H7	1.001
C1	C2	1.21(1)	C16	C17	1.34(1)
C2	C3	1.40(1)	C16	H8	0.985
C3	C4	1.20(1)	C17	C18	1.43(1)
C4	C5	1.37(1)	C17	H9	0.976
C5	C6	1.20(1)	C18	H10	1.005
C7	C8	1.324(9)	C19	C20	1.38(1)
C7	C12	1.38(1)	C19	C24	1.39(1)
C8	C9	1.36(1)	C20	C21	1.43(1)
C8	H1	0.980	C20	H11	1.011
C9	C10	1.27(1)	C21	C22	1.35(1)
C9	H2	1.019	C21	H12	0.993
C10	C11	1.45(1)	C22	C23	1.33(1)
C10	H3	1.013	C22	H13	0.981
C11	C12	1.42(1)	C23	C24	1.36(1)
C11	H4	0.993	C23	H14	1.003
C12	H5	0.996	C24	H15	0.985

Intramolecular Bond Angles

atom	atom	atom	angle	atom	atom	atom	angle
O1	P1	C7	112.3(3)	C12	C11	H4	120.40
O1	P1	C13	110.4(4)	C7	C12	C11	117.6(8)
O1	P1	C19	113.4(4)	C7	C12	H5	121.22
C7	P1	C13	108.8(4)	C11	C12	H5	121.21
C7	P1	C19	105.0(4)	P1	C13	C14	117.2(7)
C13	P1	C19	106.5(4)	P1	C13	C18	123.8(7)
I1	C1	C2	176.5(8)	C14	C13	C18	118.8(8)
C1	C2	C3	178(1)	C13	C14	C15	123.1(9)
C2	C3	C4	174(1)	C13	C14	H6	117.87
C3	C4	C5	178(1)	C15	C14	H6	119.04
C4	C5	C6	179(1)	C14	C15	C16	116.0(9)
I2	C6	C5	175.2(8)	C14	C15	H7	122.70
P1	C7	C8	116.3(6)	C16	C15	H7	121.28
P1	C7	C12	122.9(7)	C15	C16	C17	122(1)
C8	C7	C12	120.6(8)	C15	C16	H8	117.94
C7	C8	C9	121.5(9)	C17	C16	H8	119.58
C7	C8	H1	118.25	C16	C17	C18	119(1)
C9	C8	H1	120.19	C16	C17	H9	120.63
C8	C9	C10	123(1)	C18	C17	H9	120.77
C8	C9	H2	115.82	C13	C18	C17	120.9(8)
C10	C9	H2	120.73	C13	C18	H10	118.83
C9	C10	C11	119.0(9)	C17	C18	H10	120.27
C9	C10	H3	121.52	P1	C19	C20	116.1(6)
C11	C10	H3	118.79	P1	C19	C24	123.4(7)
C10	C11	C12	117.9(8)	C20	C19	C24	120.5(8)
C10	C11	H4	121.72	C19	C20	C21	117.0(8)

Angles are in degrees. Estimated standard deviations in the least significant figure are given in parentheses.

Intramolecular Bond Angles

atom	atom	atom	angle
C19	C20	H11	120.47
C21	C20	H11	122.55
C20	C21	C22	119(1)
C20	C21	H12	120.65
C22	C21	H12	120.03
C21	C22	C23	123(1)
C21	C22	H13	118.47
C23	C22	H13	118.90
C22	C23	C24	120(1)
C22	C23	H14	120.72
C24	C23	H14	119.50
C19	C24	C23	120.2(9)
C19	C24	H15	119.05
C23	C24	H15	120.63

cont

atom	atom	atom	angle

Intermolecular Distances

atom	atom	distance	ADC(*)	atom	atom	distance	ADC(*)
I1	O1	2.759(5)	76603	C5	H3	2.674	75603
I1	H11	3.382	55401	C5	H1	3.538	45404
I1	H1	3.382	76603	C5	C10	3.57(1)	75603
I1	H8	3.388	65602	C6	H3	2.669	75603
I1	H12	3.521	55401	C6	C10	3.57(1)	75603
I1	H13	3.578	75602	C7	H13	3.451	45404
I2	O1	2.863(5)	64602	C7	H12	3.467	45404
I2	H11	3.307	64602	C8	H13	2.958	45404
C1	H8	2.841	65602	C9	H13	3.106	45404
C1	H11	3.286	55401	C9	H9	3.251	75703
C1	H12	3.311	55401	C10	H9	3.202	75703
C1	H9	3.364	65602	C10	H12	3.554	45404
C2	H7	3.047	55404	C10	H13	3.594	45404
C2	H8	3.187	65602	C11	H10	3.110	75703
C2	H6	3.230	55404	C11	H12	3.255	45404
C2	H2	3.507	45404	C11	H9	3.565	75703
C2	H9	3.524	65602	C12	H12	3.161	45404
C3	H7	2.820	55404	C12	H10	3.268	75703
C3	H6	3.202	55404	C15	H14	3.288	45501
C3	H1	3.407	45404	C15	H8	3.455	65703
C3	H2	3.517	45404	C15	H14	3.570	75703
C3	C15	3.57(1)	55404	C16	H14	3.318	45501
C4	H7	3.054	55404	C16	H14	3.519	75703
C4	H1	3.237	45404	C16	H7	3.585	65703
C4	H3	3.254	75603	C17	H15	3.080	75703
C4	H6	3.544	55404	C18	H15	3.065	75703

Contacts out to 3.60 angstroms. Estimated standard deviations in the least significant figure are given in parentheses.

Intermolecular Distances

cont

atom	atom	distance	ADC(*)	atom	atom	distance	ADC(*)
C23	H5	3.009	75703				
C23	H7	3.161	65501				
C23	H8	3.566	65501				
C24	H5	3.243	75703				
H1	H13	3.102	45404				
H2	H13	3.255	45404				
H2	H9	3.584	75703				

H3	H9	3.343	75703
H4	H10	3.139	75703
H5	H14	2.759	75703
H5	H15	3.166	75703
H5	H12	3.446	45404
H5	H10	3.455	75703
H7	H14	2.647	45501
H7	H8	3.065	65703
H8	H14	2.700	45501
H8	H13	3.490	45501
H9	H15	3.195	75703
H10	H15	3.195	75703

Contacts out to 3.60 angstroms. Estimated standard deviations in the least significant figure are given in parentheses.

#===END