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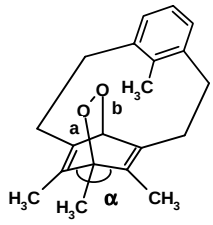
Table 1 Fractional atomic coordinates and equivalent isotropic displacement parameters ($Å^2$) for AAO_5O_5 (**3**)

Atom	x/a	y/b	z/c	Beq
C(1)	0.2540(2)	-0.1440(4)	0.2256(1)	3.6(1)
C(2)	0.1946(2)	-0.1517(4)	0.1573(1)	3.5(1)
C(3)	0.1111(2)	-0.2733(5)	0.1281(2)	4.5(2)
C(4)	0.0575(2)	-0.2794(5)	0.0614(2)	5.2(2)
C(5)	0.0834(2)	-0.1641(5)	0.0202(2)	5.2(2)
C(6)	0.1592(2)	-0.0413(5)	0.0457(2)	4.4(1)
C(7)	0.2193(2)	-0.0284(4)	0.1160(1)	3.5(1)
C(8)	0.2976(2)	0.0971(4)	0.1449(1)	3.6(1)
C(9)	0.3567(2)	0.1061(4)	0.2135(1)	3.4(1)
C(10)	0.4390(2)	0.2305(5)	0.2439(1)	4.2(2)
C(11)	0.4920(2)	0.2369(5)	0.3104(2)	5.0(2)
C(12)	0.4669(2)	0.1173(5)	0.3511(2)	5.1(2)
C(13)	0.3915(2)	-0.0062(5)	0.3249(1)	4.2(2)
C(14)	0.3333(2)	-0.0200(2)	0.2546(1)	3.5(1)
O(30)	0.2302(1)	-0.2610(3)	0.2660(1)	4.2(1)
C(31)	0.2733(2)	-0.4464(5)	0.2728(2)	5.0(2)
C(32)	0.2249(2)	-0.5718(5)	0.3025(2)	5.3(2)
O(33)	0.1210(2)	-0.5983(3)	0.2643(1)	5.7(1)
C(34)	0.0971(2)	-0.7430(5)	0.2185(2)	6.3(2)
C(35)	-0.0125(3)	-0.7612(5)	0.1810(2)	6.6(2)
O(36)	-0.0525(2)	-0.6006(3)	0.1405(1)	6.1(1)
C(37)	-0.1565(3)	-0.5908(6)	0.1162(2)	6.1(2)
C(38)	-0.1937(3)	-0.4054(6)	0.0850(2)	6.5(1)
O(39)	-0.1912(2)	-0.3778(3)	0.0237(1)	5.3(1)
C(40)	-0.2672(2)	-0.4737(5)	-0.0297(1)	4.6(2)
C(41)	-0.2713(2)	-0.3932(5)	-0.0926(2)	4.5(2)
O(42)	-0.3211(1)	-0.2148(3)	-0.1045(2)	4.0(1)
C(43)	0.4143(2)	-0.0074(6)	0.0090(2)	6.4(2)
C(44)	0.4295(3)	-0.1289(6)	-0.0330(2)	7.2(2)
C(45)	0.5142(2)	-0.1202(6)	-0.0419(2)	6.4(2)

Table 2 Fractional atomic coordinates and equivalent isotropic displacement parameters ($Å^2$) for AAO_5O_5 di-endoperoxide (**4**)

Atom	x/a	y/b	z/c	Beq
C(1)	0.653(1)	0.1719(8)	0.8498(8)	4.0(7)
C(2)	0.523(1)	0.1869(8)	0.9096(7)	3.2(6)
C(3)	0.526(1)	0.2495(8)	1.0306(8)	4.4(7)
C(4)	0.407(1)	0.2434(8)	1.0747(8)	4.4(7)
C(5)	0.286(1)	0.1664(9)	0.9924(8)	5.2(8)
C(6)	0.280(1)	0.1030(9)	0.8708(8)	4.7(6)
C(7)	0.401(1)	0.1085(8)	0.8292(7)	3.0(7)
C(8)	0.425(1)	0.0392(8)	0.7037(8)	3.6(8)
C(9)	0.604(1)	-0.0383(7)	0.7216(7)	2.9(9)
C(10)	0.649(1)	-0.1669(8)	0.6686(8)	3.9(7)
C(11)	0.821(1)	-0.2223(8)	0.7028(8)	4.4(7)
C(12)	0.944(1)	-0.1530(8)	0.7817(8)	4.4(7)
C(13)	0.897(1)	-0.0213(8)	0.8333(8)	3.3(6)
C(14)	0.729(1)	0.0349(7)	0.8044(7)	2.5(6)
O(15)	0.5475(8)	0.2165(6)	0.7329(5)	4.5(4)
O(16)	0.4058(8)	0.1457(6)	0.6506(5)	4.5(5)
O(30)	0.7748(7)	0.2482(5)	0.9171(5)	4.2(4)
C(31)	0.719(1)	0.3834(8)	0.9256(8)	4.5(7)
C(32)	0.829(1)	0.4553(9)	1.0457(9)	5.2(8)
O(53)	0.2315(9)	-0.4677(6)	0.8585(6)	6.5(6)
C(54)	0.164(1)	-0.5524(9)	0.7459(8)	5.9(8)
C(55)	0.239(1)	-0.5506(8)	0.6540(9)	5.3(8)
O(56)	0.188(1)	-0.4281(6)	0.6343(7)	7.9(6)
C(57)	0.269(1)	-0.4160(9)	0.5579(9)	6.5(9)
C(58)	0.193(1)	-0.2841(9)	0.5422(9)	6.8(9)
O(59)	0.2487(8)	-0.2736(6)	0.4543(5)	5.5(5)
C(60)	0.163(1)	-0.1588(9)	0.4185(8)	4.6(7)
C(61)	0.274(1)	-0.0575(9)	0.4885(8)	4.9(7)
O(62)	0.2819(7)	-0.0144(6)	0.6189(5)	4.4(5)



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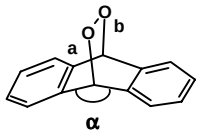

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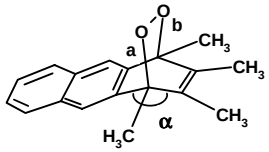

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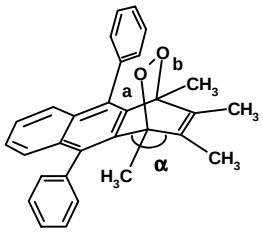

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Diagram 5

The O-O bond lengths of **7**-**10** are between 147 and 150 pm within experimental errors. For **4**, the value found 149 pm is equal to that measured for O-O in H_2O_2;[25] this is the result of several factors (strain, electronic density) difficult to analyze. Regarding the C-O bond lengths, they are strikingly longer than the tabulated average data known to be *ca* 142 pm.[26] In Table 3 they span 148-152 pm and reflect a weaker bonding; compound **4** appears to have the weakest bonds (within experimental errors) and at least weaker than those of **8**. The angle α does not experience significant changes within the series.

Table 3 Selected bond lengths and angles of compound **4**, in comparison with those of other 1,4-endoperoxides. (1) and (2) refer to the two independent molecules in the unit cell; α_1 and α_2 are values of the two α angles due non-symmetrical molecular geometry.

compound	bond lengths (pm)			angle (α)°	reference
	O-O	C-O (a)	C-O (b)		
7	147.0	149.8	149.7	$\alpha_1 \approx \alpha_2 \approx 111$	23
8	147.6	147.6	147.8	107	24
9	(1) 148.6	148.8	150.2	α_1 111, α_2 108	10c
	(2) 148.9	148.9	149.5	α_1 106, α_2 107	
10	149.8	142.6	148.2	α_1 108, α_2 104	10c
4	149.0	151.0	152.0	α_1 111, α_2 110	this study

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