Supporting Information

trans-2,2'-Bi(1-phenyladamantylidene): The Most Twisted Biadamantylidene.

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Table S1. Electronic Energies (E), Zero Point Energies (ZPE) and Gibbs Free Energies (G) Obtained from HF and DFT Calculations

Olefin	Theory/Basis	E, hartree	ZPE, hartree	G, hartree	Number of imaginary frequencies
1-H	HF/6-31G(d)	-773.7016584	0.476923	-773.263159	0
	BLYP/6-31G(d)	-778.5357152	0.432364	-778.142814	0
1-Et	HF/6-31G(d)	-929.7856565	0.598945	-929.231933	0
	BLYP/6-31G(d)	-935.6360066	0.542715	-935.139042	0
1-Ph	HF/6-31G(d)	-1232.7471648	0.650259	-1232.145542	0
	BLYP/6-31G(d)	-1240.3919246	0.588413	-1239.854302	0

Table S2. Cartesian Coordinates for Adamantylideneadamantane (1-H) Optimized at BLYP/6-31G(d) Level

Center Number	Atomic Number	Atomic Type	Coordinates (Angstroms)		
			X	Y	Z
1	6	0	0.680638	0.000000	0.000000
2	6	0	-0.680638	0.000000	0.000000
3	6	0	-1.561408	1.260426	0.000000
4	6	0	-1.561408	-1.260426	0.000000
5	6	0	1.561408	1.260426	0.000000
6	6	0	1.561408	-1.260426	0.000000
7	6	0	-4.268698	0.000000	0.000000
8	6	0	4.268698	0.000000	0.000000
9	1	0	-4.928750	0.888213	0.000000
10	1	0	-4.928750	-0.888213	0.000000
11	1	0	-0.952108	2.176709	0.000000
12	1	0	-0.952108	-2.176709	0.000000
13	1	0	0.952108	2.176709	0.000000
14	1	0	0.952108	-2.176709	0.000000
15	1	0	4.928750	0.888213	0.000000
16	1	0	4.928750	-0.888213	0.000000
17	6	0	-3.374193	0.000000	1.272797
18	6	0	-3.374193	0.000000	-1.272797
19	6	0	3.374193	0.000000	1.272797
20	6	0	3.374193	0.000000	-1.272797
21	1	0	-4.013609	0.000000	-2.175321
22	1	0	-4.013609	0.000000	2.175321
23	1	0	4.013609	0.000000	-2.175321
24	1	0	4.013609	0.000000	2.175321
25	6	0	2.471724	1.263397	1.270592
26	6	0	-2.471724	-1.263397	-1.270592
27	6	0	2.471724	-1.263397	1.270592
28	6	0	2.471724	1.263397	-1.270592
29	6	0	-2.471724	1.263397	1.270592
30	6	0	-2.471724	-1.263397	1.270592
31	6	0	2.471724	1.263397	-1.270592
32	6	0	-2.471724	1.263397	-1.270592
33	1	0	-1.845503	1.288495	-2.181125
34	1	0	-1.845503	-1.288495	-2.181125
35	1	0	-1.845503	1.288495	2.181125
36	1	0	-1.845503	-1.288495	2.181125
37	1	0	1.845503	1.288495	-2.181125
38	1	0	1.845503	-1.288495	-2.181125
39	1	0	1.845503	1.288495	2.181125
40	1	0	1.845503	-1.288495	2.181125
41	1	0	-3.093839	2.178789	-1.278090
42	1	0	-3.093839	-2.178789	-1.278090
43	1	0	-3.093839	2.178789	1.278090
44	1	0	-3.093839	-2.178789	1.278090
45	1	0	3.093839	2.178789	-1.278090
46	1	0	3.093839	-2.178789	-1.278090
47	1	0	3.093839	2.178789	1.278090
48	1	0	3.093839	-2.178789	1.278090

Table S3. Cartesian Coordinates for Adamantylideneadamantane (1-H) Optimized at HF/6-31G(d) Level

Center Number	Atomic Number	Atomic Type	Coordinates (Angstroms)		
			X	Y	Z
1	6	0	0.666033	0.000000	0.000000
2	6	0	-0.666033	0.000000	0.000000
3	6	0	-1.544498	1.249428	0.000000
4	6	0	-1.544498	-1.249428	0.000000
5	6	0	1.544498	1.249428	0.000000
6	6	0	1.544498	-1.249428	0.000000
7	6	0	-4.217837	0.000000	0.000000
8	6	0	4.217837	0.000000	0.000000
9	1	0	-4.866566	0.873443	0.000000
10	1	0	-4.866566	-0.873443	0.000000
11	1	0	-0.951101	2.151615	0.000000
12	1	0	-0.951101	-2.151615	0.000000
13	1	0	0.951101	2.151615	0.000000
14	1	0	0.951101	-2.151615	0.000000
15	1	0	4.866566	0.873443	0.000000
16	1	0	4.866566	-0.873443	0.000000
17	6	0	-3.333606	0.000000	1.257856
18	6	0	-3.333606	0.000000	-1.257856
19	6	0	3.333606	0.000000	1.257856
20	6	0	3.333606	0.000000	-1.257856
21	1	0	-3.961325	0.000000	-2.145597
22	1	0	-3.961325	0.000000	2.145597
23	1	0	3.961325	0.000000	-2.145597
24	1	0	3.961325	0.000000	2.145597
25	6	0	2.440448	1.247744	1.254708
26	6	0	-2.440448	-1.247744	-1.254708
27	6	0	2.440448	-1.247744	1.254708
28	6	0	2.440448	1.247744	-1.254708
29	6	0	-2.440448	1.247744	1.254708
30	6	0	-2.440448	-1.247744	1.254708
31	6	0	2.440448	1.247744	-1.254708
32	6	0	-2.440448	1.247744	-1.254708
33	1	0	-1.823117	1.270279	-2.149348
34	1	0	-1.823117	-1.270279	-2.149348
35	1	0	-1.823117	1.270279	2.149348
36	1	0	-1.823117	-1.270279	2.149348
37	1	0	1.823117	1.270279	-2.149348
38	1	0	1.823117	-1.270279	-2.149348
39	1	0	1.823117	1.270279	2.149348
40	1	0	1.823117	-1.270279	2.149348
41	1	0	-3.052081	2.147827	-1.266801
42	1	0	-3.052081	-2.147827	-1.266801
43	1	0	-3.052081	2.147827	1.266801
44	1	0	-3.052081	-2.147827	1.266801
45	1	0	3.052081	2.147827	-1.266801
46	1	0	3.052081	-2.147827	-1.266801
47	1	0	3.052081	2.147827	1.266801
48	1	0	3.052081	-2.147827	1.266801

Table S4. Cartesian Coordinates for trans-Bis(1-phenyladamantylidene) (1-Ph) Optimized at BLYP/6-31G(d) Level

Center Number	Atomic Number	Atomic Type	Coordinates (Angstroms)		
			X	Y	Z
1	6	0	-0.411890	0.548506	-0.244327
2	6	0	0.411890	-0.548506	-0.244327
3	6	0	0.088576	1.958260	-0.626070
4	6	0	-0.088576	-1.958260	-0.626070
5	6	0	-1.984299	0.544044	-0.147582
6	6	0	1.984299	-0.544044	-0.147582
7	6	0	-2.641731	-0.704713	0.489345
8	6	0	2.641731	0.704713	0.489345
9	6	0	-0.448247	2.235261	-2.073148
10	6	0	0.448247	-2.235261	-2.073148
11	6	0	-2.543531	0.886938	-1.574779
12	6	0	2.543531	-0.886938	-1.574779
13	6	0	-2.436309	1.736240	0.802495
14	6	0	2.436309	-1.736240	0.802495
15	6	0	-0.403070	3.097417	0.307846
16	6	0	0.403070	-3.097417	0.307846
17	6	0	-1.996153	2.255441	-2.067479
18	6	0	1.996153	-2.255441	-2.067479
19	6	0	-1.948652	3.116898	0.310840
20	6	0	1.948652	-3.116898	0.310840
21	6	0	-2.487025	3.383876	-1.120211
22	6	0	2.487025	-3.383876	-1.120211
23	6	0	-2.246189	-1.120705	1.785570
24	6	0	2.246189	1.120705	1.785570
25	6	0	-3.722796	-1.391976	-0.108454
26	6	0	3.722796	1.391976	-0.108454
27	6	0	-4.370576	-2.453955	0.551213
28	6	0	4.370576	2.453955	0.551213
29	6	0	-2.891449	-2.176044	2.447915
30	6	0	2.891449	2.176044	-2.447915
31	6	0	-3.961114	-2.851634	1.832826
32	6	0	3.961114	2.851634	1.832826
33	1	0	1.181558	1.976373	-0.668560
34	1	0	-1.181558	-1.976373	-0.668560
35	1	0	-2.372049	2.440273	-3.091314
36	1	0	2.372049	-2.440273	-3.091314
37	1	0	-2.319686	3.897898	1.001194
38	1	0	2.319686	-3.897898	1.001194
39	1	0	-0.055478	3.207976	-2.426746
40	1	0	0.055478	-3.207976	-2.426746
41	1	0	-0.074554	1.461185	-2.767963
42	1	0	0.074554	-1.461185	-2.767963
43	1	0	-0.016635	2.945408	1.330860
44	1	0	0.016635	-2.945408	1.330860
45	1	0	0.000000	4.064407	-0.048284
46	1	0	0.000000	-4.064407	-0.048284
47	1	0	-3.540220	1.713256	0.859009
48	1	0	3.540220	-1.713256	0.859009
49	1	0	-2.064710	1.553768	1.825389
50	1	0	2.064710	-1.553768	1.825389
51	1	0	-2.273944	0.087696	-2.288912
52	1	0	2.273944	-0.087696	-2.288912
53	1	0	-3.647553	0.938146	-1.543136
54	1	0	3.647553	-0.938146	-1.543136
55	1	0	-3.593008	3.421317	-1.111494
56	1	0	3.593008	-3.421317	-1.111494
57	1	0	-2.134246	4.366708	-1.485509
58	1	0	2.134246	-4.366708	-1.485509
59	1	0	-1.409933	-0.615195	2.275733
60	1	0	1.409933	0.615195	2.275733
61	1	0	-2.557996	-2.470791	3.448123

62	1	0	2.557996	2.470791	3.448123
63	1	0	-4.466315	-3.674824	2.346862
64	1	0	4.466315	3.674824	2.346862
65	1	0	-5.200731	-2.967363	0.055604
66	1	0	5.200731	2.967363	0.055604
67	1	0	-4.065815	-1.113696	-1.106859
68	1	0	4.065815	1.113696	-1.106859

Table S5. Cartesian Coordinates for trans-Bis(1-phenyladamantylidene) (1-Ph) Optimized at HF/6-31G(d) Level

Center Number	Atomic Number	Atomic Type	Coordinates (Angstroms)		
			X	Y	Z
1	6	0	0.666206	0.082354	-0.236912
2	6	0	-0.666206	-0.082354	-0.236912
3	6	0	1.633569	-1.045164	-0.623147
4	6	0	-1.633569	1.045164	-0.623147
5	6	0	1.450418	1.431058	-0.140723
6	6	0	-1.450418	-1.431058	-0.140723
7	6	0	0.715093	2.621196	0.500326
8	6	0	-0.715093	-2.621196	0.500326
9	6	0	2.121645	-0.714427	-2.052334
10	6	0	-2.121645	0.714427	-2.052334
11	6	0	2.005082	1.744986	-1.552807
12	6	0	-2.005082	-1.744986	-1.552807
13	6	0	2.694212	1.219763	0.785115
14	6	0	-2.694212	-1.219763	0.785115
15	6	0	2.857399	-1.187481	0.289666
16	6	0	-2.857399	1.187481	0.289666
17	6	0	2.902052	0.601992	-2.052571
18	6	0	-2.902052	-0.601992	-2.052571
19	6	0	3.636777	0.125877	0.288298
20	6	0	-3.636777	-0.125877	0.288298
21	6	0	4.118758	0.461383	-1.128146
22	6	0	-4.118758	-0.461383	-1.128146
23	6	0	0.185305	2.496389	1.787094
24	6	0	-0.185305	-2.496389	1.787094
25	6	0	0.635797	3.872685	-0.099698
26	6	0	-0.635797	-3.872685	-0.099698
27	6	0	0.046875	4.951208	0.549450
28	6	0	-0.046875	-4.951208	0.549450
29	6	0	-0.398239	3.566432	2.437378
30	6	0	0.398239	-3.566432	2.437378
31	6	0	-0.471875	4.807199	1.820506
32	6	0	0.471875	-4.807199	1.820506
33	1	0	1.119362	-1.985516	-0.666813
34	1	0	-1.119362	1.985516	-0.666813
35	1	0	3.233750	0.833766	-3.061811
36	1	0	-3.233750	-0.833766	-3.061811
37	1	0	4.488776	0.058297	0.960775
38	1	0	-4.488776	-0.058297	0.960775
39	1	0	2.755661	-1.522916	-2.411241
40	1	0	-2.755661	1.522916	-2.411241
41	1	0	1.272982	-0.652063	-2.727836
42	1	0	-1.272982	0.652063	-2.727836
43	1	0	2.549958	-1.441597	1.298782
44	1	0	-2.549958	1.441597	1.298782
45	1	0	3.478425	-2.006382	-0.067279
46	1	0	-3.478425	2.006382	-0.067279
47	1	0	3.218985	2.169284	0.849476
48	1	0	-3.218985	-2.169284	0.849476
49	1	0	2.367013	0.979287	1.790707
50	1	0	-2.367013	-0.979287	1.790707
51	1	0	1.186548	1.908452	-2.248834
52	1	0	-1.186548	-1.908452	-2.248834
53	1	0	2.592740	2.658043	-1.526864
54	1	0	-2.592740	-2.658043	-1.526864
55	1	0	4.693969	1.384855	-1.119269
56	1	0	-4.693969	-1.384855	-1.119269
57	1	0	4.780402	-0.319554	-1.496409
58	1	0	-4.780402	0.319554	-1.496409
59	1	0	0.218850	1.543814	2.282322
60	1	0	-0.218850	-1.543814	2.282322
61	1	0	-0.796937	3.432543	3.427778

62	1	0	0.796937	-3.432543	3.427778
63	1	0	-0.925522	5.641682	2.324745
64	1	0	0.925522	-5.641682	2.324745
65	1	0	0.000000	5.903940	0.051740
66	1	0	0.000000	-5.903940	0.051740
67	1	0	1.019867	4.028073	-1.088932
68	1	0	-1.019867	-4.028073	-1.088932

Table S6. Cartesian Coordinates for trans-Bis(1-ethyladamantylidene) (1-Et) Optimized at BLYP/6-31G(d) Level

Center Number	Atomic Number	Atomic Type	Coordinates (Angstroms)		
			X	Y	Z
1	6	0	1.746548	1.122201	0.198227
2	6	0	0.687000	-0.043581	0.065085
3	6	0	1.421012	-1.341138	-0.361477
4	6	0	2.597925	-1.784689	0.549612
5	6	0	3.633992	-0.641518	0.614019
6	6	0	4.207781	-0.356887	-0.800263
7	6	0	3.051259	0.060914	-1.749264
8	6	0	2.384909	1.357580	-1.217042
9	6	0	2.905836	0.611811	1.147634
10	6	0	1.996341	-1.072040	-1.798297
11	6	0	1.325919	2.511613	0.789875
12	6	0	0.696984	2.516210	2.202444
13	6	0	-0.687029	0.043571	0.065075
14	6	0	-1.746596	-1.122184	0.198349
15	6	0	-1.421034	1.341102	-0.361593
16	6	0	-2.385039	-1.357643	-1.216872
17	6	0	-2.905812	-0.611673	1.147781
18	6	0	-1.325966	-2.511606	0.789994
19	6	0	-2.597886	1.784776	0.549519
20	6	0	-1.996444	1.071894	-1.798351
21	6	0	-3.051394	-0.061016	-1.749164
22	6	0	-3.633968	0.641629	0.614096
23	6	0	-0.696575	-2.516215	2.202354
24	6	0	-4.207851	0.356888	-0.800126
25	1	0	-3.450914	-0.244918	-2.764235
26	1	0	-4.456644	0.909659	1.303550
27	1	0	-0.710897	2.164207	-0.457570
28	1	0	0.710912	-2.164288	-0.457331
29	1	0	3.450711	0.244744	-2.764375
30	1	0	4.456713	-0.909466	1.303452
31	1	0	-4.717023	1.257085	-1.193089
32	1	0	-4.967791	-0.445822	-0.749345
33	1	0	-2.453459	2.004457	-2.181941
34	1	0	-1.174534	0.804873	-2.487014
35	1	0	-1.606908	-1.707034	-1.920861
36	1	0	-3.140736	-2.165190	-1.150005
37	1	0	-3.629221	-1.440003	1.270521
38	1	0	-2.497591	-0.395914	2.150739
39	1	0	-2.238363	2.030160	1.564045
40	1	0	-3.053335	2.706325	0.138881
41	1	0	-2.254567	-3.112956	0.829635
42	1	0	-0.676462	-3.062393	0.091673
43	1	0	2.254461	3.113077	0.829180
44	1	0	0.676116	3.062272	0.091731
45	1	0	3.629218	1.440183	1.270232
46	1	0	2.497686	0.396149	2.150644
47	1	0	1.606745	1.706902	-1.921028
48	1	0	3.140602	2.165141	-1.150276
49	1	0	4.967709	0.445842	-0.749596
50	1	0	4.716943	-1.257103	-1.193194
51	1	0	2.453380	-2.004614	-2.181832
52	1	0	1.174381	-0.805112	-2.486932
53	1	0	-2.238463	-2.029978	1.564187
54	1	0	3.053364	-2.706273	0.139044
55	1	0	1.421316	2.200266	2.971005
56	1	0	0.361456	3.533095	2.469156
57	1	0	-0.170358	1.842989	2.270756
58	1	0	-1.420589	-2.200042	2.971117
59	1	0	-0.361191	-3.533152	2.469051
60	1	0	0.170941	-1.843185	2.270328

Table S7. Cartesian Coordinates for trans-Bis(1-ethyladamantylidene) (1-Et) Optimized at HF/6-31G(d) Level

Center Number	Atomic Number	Atomic Type	Coordinates (Angstroms)		
			X	Y	Z
1	6	0	-1.743618	1.130572	-0.091995
2	6	0	-0.673149	-0.031636	-0.081090
3	6	0	-1.424133	-1.351511	0.175557
4	6	0	-2.557453	-1.659108	-0.813603
5	6	0	-3.591648	-0.543486	-0.751290
6	6	0	-4.186303	-0.447290	0.659918
7	6	0	-3.063752	-0.166191	1.668813
8	6	0	-2.386130	1.162415	1.320336
9	6	0	-2.865750	0.751057	-1.105008
10	6	0	-2.025089	-1.285995	1.596203
11	6	0	-1.376484	2.594590	-0.447093
12	6	0	-0.790450	2.847178	-1.840844
13	6	0	0.673178	0.031699	-0.081098
14	6	0	1.743589	-1.130554	-0.092035
15	6	0	1.424218	1.351535	0.175583
16	6	0	2.557113	-1.162460	1.320294
17	6	0	2.865726	-0.751073	-1.105053
18	6	0	1.376351	-2.594535	-0.447179
19	6	0	2.557538	1.659117	-0.813576
20	6	0	2.025196	1.285954	1.596221
21	6	0	3.063811	0.166105	1.668785
22	6	0	3.591681	0.543435	-0.751317
23	6	0	0.790252	-2.847003	-1.840926
24	6	0	4.186360	0.447173	0.659872
25	1	0	3.478491	0.107271	2.672338
26	1	0	4.385250	0.721539	-1.473339
27	1	0	0.744593	2.171638	0.157268
28	1	0	-0.744476	-2.171587	0.157218
29	1	0	-3.478419	-0.107401	2.672375
30	1	0	-4.385220	-0.721599	-1.473306
31	1	0	4.696806	1.373579	0.914704
32	1	0	4.931896	-0.344425	0.698883
33	1	0	2.483953	2.243062	1.837960
34	1	0	1.236286	1.117539	2.324428
35	1	0	1.623968	-1.400834	2.058438
36	1	0	3.121698	-1.963448	1.358841
37	1	0	3.580296	-1.570099	-1.137826
38	1	0	2.445134	-0.663292	-2.102242
39	1	0	2.173786	1.749391	-1.825147
40	1	0	3.001020	2.619438	-0.557377
41	1	0	2.308075	-3.149711	-0.381617
42	1	0	0.754021	-3.037724	0.318018
43	1	0	-2.308247	3.149694	-0.381476
44	1	0	-0.754171	3.037800	0.318108
45	1	0	-3.580361	1.570047	-1.137759
46	1	0	-2.445161	0.663310	-2.102202
47	1	0	-1.623995	1.400814	2.058480
48	1	0	-3.121757	1.963365	1.358901
49	1	0	-4.931876	0.344273	0.698965
50	1	0	-4.696698	-1.373723	0.914752
51	1	0	-2.483804	-2.243129	1.837924
52	1	0	-1.236170	-1.117566	2.324395
53	1	0	-2.173700	-1.749316	-1.825182
54	1	0	-3.000893	-2.619460	-0.557448
55	1	0	-1.555495	2.777123	-2.605949
56	1	0	-0.368848	3.846299	-1.899886
57	1	0	-0.012810	2.140622	-2.095997
58	1	0	1.555298	-2.776996	-2.606036
59	1	0	0.368542	-3.846076	-1.900001
60	1	0	0.012680	-2.140354	-2.096045

06-JUL-00 16:24:56

¹H NMR (270 Mz)in CDCl₃ at rt

OFR 270.05 MHz

r.t., CDCl₃

EXMOD	NON
SLVNT	CDCl ₃
POINT	32768
OBSET	112.00 kHz
OBFIN	5800.0 Hz
IRSET	112.00 kHz
IRFIN	5800.0 Hz
IRATN	511
PW1	5.7 us
ACQTM	3.031 sec
PD	1.969 sec
SCANS	16
BF	0.10 Hz
OPERATOR :

Figure S1. ¹H NMR spectrum for 1-Ph.

67.80 MHz
OFR $\text{r.t.}, \text{CDCl}_3$

EXMOD BCM

SLVNT CDCL3

POINT 65536

OBSET 135.00 KHZ

08F IN 5200.0 Hz

IRASET 112.00 kHz

IRF IN 5800.0 Hz

IRATN 511

PW1	3.9 us
JUL	

ACGTM 1.638 sec

PD 1.362 sec

SCANS 1479

1.00 Hz

OPERATOR :

Figure S2. ^{13}C NMR spectrum for 1-Ph.

14-DEC-00 14:20:04
¹H NMR (270 Mz)
 in CDCl₃ at rt

OFR 270.05 MHz

rt., CDCl₃

EXMOD NON
 SLVNT CDCL3
 POINT 32768
 OBSET 112.00 KHz
 OBFIN 5800.0 Hz
 IRSET 112.00 KHz
 IRFIN 5800.0 Hz
 IRATN 511
 PW1 5.7 us
 ACQTM 3.031 sec
 PD 1.969 sec
 SCANS 32
 BF 0.10 Hz
 OPERATOR :

Figure S3. ¹H NMR spectrum for 7.

25-NOV-00 01:01:48

¹³C NMR (67.8 Mz)
in CDCl₃ at rt

OFR 100.40 MHz

rt., CDCl₃

EXMOD BCM
SLVNT CDCL3
POINT 65536
OBSET 125.00 KHZ
OBFIN 10500.0 HZ
IRSET 124.00 KHZ
IRFIN 10500.0 HZ
IRATN 511
PW1 4.7 us
ACQTM 1.088 sec
PD 1.912 sec
SCANS 6000
BF 1.00 HZ
OPERATOR :

Figure S4. ¹³C NMR spectrum for 7.

21-OCT-00 10:16:56

00102141

OFR

399.65 MHz

¹H NMR (400 Mz)
in CDCl₃ at rt

EXMOD NON
SLVNT CDCL3
POINT 32768
OBSET 124.00 kHz
OBFIN 10500.0 Hz
IRSET 124.00 kHz
IRFIN 10500.0 Hz
IRATN 511
PW1 6.5 us
ACQTM 2.048 sec
PD 2.952 sec
SCANS 32
BF 0.10 Hz
OPERATOR :

Figure S5. ¹H NMR spectrum for 10.

21-OCT-00 10:06:18

QFR 100.40 MHz

^{13}C NMR (100 Mz)
in CDCl_3 at rt

EXMOD BCM
SLVNT CDCl_3
POINT 65536
OBSET 125.00 kHz
OBFIN 10500.0 Hz
IRSET 124.00 kHz
IRFIN 10500.0 Hz
IRATN 511
PW1 4.7 us
ACQTM 1.088 sec
PD 1.912 sec
SCANS 16515
-BF 1.00 Hz
OPERATOR :

Figure S6. ^{13}C NMR spectrum for 10.

X-ray Structure Report

for 1-Ph

Mon Jan 17 2000

Experimental

Data Collection

A colorless prismatic crystal of $C_{32}H_{36}$ having approximate dimensions of 0.20 x 0.20 x 0.20 mm was mounted on a glass fiber. All measurements were made on a Rigaku AFC7R diffractometer with graphite monochromated Mo-K α radiation and a rotating anode generator.

Cell constants and an orientation matrix for data collection, obtained from a least-squares refinement using the setting angles of 25 carefully centered reflections in the range $27.89 < 2\theta < 29.82^\circ$ corresponded to a C-centered monoclinic cell with dimensions:

$$\begin{aligned} a &= 19.768(4) \text{ \AA} \\ b &= 6.721(8) \text{ \AA} \quad \beta = 110.60(2)^\circ \\ c &= 18.437(5) \text{ \AA} \\ V &= 2292(2) \text{ \AA}^3 \end{aligned}$$

For $Z = 4$ and F.W. = 420.64, the calculated density is 1.22 g/cm³. Based on the systematic absences of:

$$\begin{aligned} hkl: h+k &\neq 2n \\ h0l: l &\neq 2n \end{aligned}$$

packing considerations, a statistical analysis of intensity distribution, and the successful solution and refinement of the structure, the space group was determined to be:

$$C2/c \text{ (\#15)}$$

The data were collected at a temperature of $23 \pm 1^\circ\text{C}$ using the ω - 2θ scan technique to a maximum 2θ value of 55.0° . Omega scans of several intense reflections, made prior to data collection, had an average width at half-height of 0.20° with a take-off angle of 6.0° . Scans of $(1.73 + 0.30 \tan \theta)^\circ$ were made at a speed of $8.0^\circ/\text{min}$ (in omega). The weak reflections ($I < 10.0\sigma(I)$) were rescanned (maximum of 2 scans) and the counts were accumulated to ensure good counting statistics. Stationary background counts were recorded on each side of the reflection. The ratio of peak counting time to background counting time was 2:1. The diameter of the incident beam collimator was 0.5 mm and the crystal to detector distance was 235 mm. The computer-controlled slits were set to 3.0 mm (horizontal) and 3.0 mm (vertical).

Data Reduction

Of the 2722 reflections which were collected, 2648 were unique ($R_{\text{int}} = 0.025$). The intensities of three representative reflection were measured after every 150 reflections. No decay correction was applied.

The linear absorption coefficient, μ , for Mo-K α radiation is 0.7 cm^{-1} . Azimuthal scans of several reflections indicated no need for an absorption correction. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods¹ and expanded using Fourier techniques². The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined isotropically. The final cycle of full-matrix least-squares refinement³ was based on 1498 observed reflections ($I > 3.00\sigma(I)$) and 217 variable parameters and converged (largest parameter shift was 0.01 times its esd) with unweighted and weighted agreement factors of:

$$R = \Sigma ||Fo| - |Fc|| / \Sigma |Fo| = 0.048$$

$$R_w = \sqrt{\Sigma w(|Fo| - |Fc|)^2 / \Sigma w Fo^2} = 0.043$$

The standard deviation of an observation of unit weight⁴ was 1.04. The weighting scheme was based on counting statistics and included a factor ($p = 0.005$) to downweight the intense reflections. Plots of $\Sigma w(|Fo| - |Fc|)^2$ versus $|Fo|$, reflection order in data collection, $\sin \theta / \lambda$ and various classes of indices showed no unusual trends. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.23 and -0.15 $e^-/\text{\AA}^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber⁵. Anomalous dispersion effects were included in Fcalc⁶; the values for $\Delta f'$ and $\Delta f''$ were those of Creagh and McAuley⁷. The values for the mass attenuation coefficients are those of Creagh and Hubbel⁸. All calculations were performed using the teXsan⁹ crystallographic software package of Molecular Structure Corporation.

References

(1) SIR92: Altomare, A., Burla, M.C., Camalli, M., Cascarano, M., Giacovazzo, C., Guagliardi, A., Polidori, G., (1994), J. Appl. Cryst., 27, 435

(2) DIRDIF94: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., de Gelder, R., Israel, R. and Smits, J.M.M. (1994). The DIRDIF-94 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

(3) Least-Squares:

Function minimized: $\Sigma w(|Fo| - |Fc|)^2$

where $w = \frac{1}{\sigma^2(Fo)} = [\sigma_c^2(Fo) + \frac{p}{4} Fo^2]^{-1}$

$\sigma_c(Fo)$ = e.s.d. based on counting statistics

p = p-factor

(4) Standard deviation of an observation of unit weight:

$$\sqrt{\Sigma w(|Fo| - |Fc|)^2 / (No - Nv)}$$

where: No = number of observations

Nv = number of variables

(5) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).

(6) Ibers, J. A. & Hamilton, W. C.; *Acta Crystallogr.*, 17, 781 (1964).

(7) Creagh, D. C. & McAuley, W.J. ; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).

(8) Creagh, D. C. & Hubbell, J.H.; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200-206 (1992).

(9) teXsan: Crystal Structure Analysis Package, Molecular Structure Corporation (1985 & 1992).

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	C ₃₂ H ₃₆
Formula Weight	420.64
Crystal Color, Habit	colorless, prismatic
Crystal Dimensions	0.20 X 0.20 X 0.20 mm
Crystal System	monoclinic
Lattice Type	C-centered
No. of Reflections Used for Unit Cell Determination (2 θ range)	25 (27.9 - 29.8°)
Omega Scan Peak Width at Half-height	0.20°
Lattice Parameters	$a = 19.768(4) \text{ \AA}$ $b = 6.721(8) \text{ \AA}$ $c = 18.437(5) \text{ \AA}$ $\beta = 110.60(2)^\circ$
	$V = 2292(2) \text{ \AA}^3$
Space Group	C2/c (#15)
Z value	4
D _{calc}	1.218 g/cm ³
F ₀₀₀	912.00
$\mu(\text{MoK}\alpha)$	0.68 cm ⁻¹

B. Intensity Measurements

Diffractometer	Rigaku AFC7R
Radiation	MoK α ($\lambda = 0.71069 \text{ \AA}$) graphite monochromated

Attenuator	Zr foil (factor = 8.63)
Take-off Angle	6.0°
Detector Aperture	3.0 mm horizontal 3.0 mm vertical
Crystal to Detector Distance	235 mm
Voltage, Current	50kV, 150mA
Temperature	23.0°C
Scan Type	ω - 2θ
Scan Rate	8.0°/min (in ω) (up to 2 scans)
Scan Width	$(1.73 + 0.30 \tan \theta)^\circ$
$2\theta_{max}$	55.0°
No. of Reflections Measured	Total: 2722 Unique: 2648 ($R_{int} = 0.025$)
Corrections	Lorentz-polarization

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR92)
Refinement	Full-matrix least-squares
Function Minimized	$\Sigma w(Fo - Fc)^2$
Least Squares Weights	$w = \frac{1}{\sigma^2(Fo)} = [\sigma_c^2(Fo) + \frac{p}{4} Fo^2]^{-1}$
p-factor	0.0050
Anomalous Dispersion	All non-hydrogen atoms
No. Observations ($I > 3.00\sigma(I)$)	1498
No. Variables	217
Reflection/Parameter Ratio	6.90
Residuals: R; Rw	0.048 ; 0.043
Residuals: R1	0.048
No. of Reflections to calc R1	1498

Goodness of Fit Indicator	1.04
Max Shift/Error in Final Cycle	0.01
Maximum peak in Final Diff. Map	$0.23 \text{ e}^-/\text{\AA}^3$
Minimum peak in Final Diff. Map	$-0.15 \text{ e}^-/\text{\AA}^3$

Table 1. Atomic coordinates and B_{iso}/B_{eq}

atom	x	y	z	B_{eq}
C(1)	0.0154(1)	0.2442(4)	0.1471(1)	2.46(5)
C(2)	0.0260(1)	0.2646(4)	0.2341(1)	2.83(5)
C(3)	0.1051(1)	0.3202(4)	0.2797(1)	3.40(6)
C(4)	0.1604(1)	0.1750(5)	0.2682(2)	4.01(7)
C(5)	0.1502(1)	0.1640(5)	0.1831(2)	3.81(6)
C(6)	0.1631(2)	0.3670(5)	0.1545(2)	4.09(7)
C(7)	0.1089(2)	0.5148(4)	0.1645(2)	3.55(6)
C(8)	0.0318(1)	0.4476(4)	0.1177(2)	3.30(6)
C(9)	0.0728(1)	0.0956(4)	0.1392(2)	3.39(6)
C(10)	0.1175(2)	0.5284(4)	0.2498(2)	3.69(6)
C(11)	-0.0570(1)	0.1574(4)	0.0952(1)	2.85(5)
C(12)	-0.1036(1)	0.2560(5)	0.0307(1)	3.69(6)
C(13)	-0.1678(2)	0.1683(7)	-0.0159(2)	5.00(8)
C(14)	-0.1859(2)	-0.0183(7)	-0.0004(2)	5.49(9)
C(15)	-0.1400(2)	-0.1217(5)	0.0627(2)	4.90(8)
C(16)	-0.0765(1)	-0.0343(4)	0.1098(2)	3.74(6)
H(1)	0.114(1)	0.336(4)	0.332(1)	3.5(5)
H(2)	0.154(1)	0.044(4)	0.289(2)	4.3(6)
H(3)	0.211(2)	0.224(4)	0.301(2)	5.3(6)
H(4)	0.186(1)	0.060(4)	0.175(2)	4.9(6)
H(5)	0.159(1)	0.359(4)	0.098(2)	4.0(6)
H(6)	0.213(2)	0.414(4)	0.182(2)	4.9(6)
H(7)	0.116(1)	0.648(4)	0.146(1)	3.9(6)
H(8)	-0.005(1)	0.551(4)	0.122(1)	4.2(6)

Table 1. Atomic coordinates and B_{iso}/B_{eq} (continued)

atom	x	y	z	B_{eq}
H(9)	0.027(1)	0.436(4)	0.062(1)	3.5(5)
H(10)	0.065(1)	-0.031(4)	0.159(1)	3.5(6)
H(11)	0.064(1)	0.078(4)	0.080(2)	4.9(6)
H(12)	0.081(1)	0.627(4)	0.257(1)	4.5(6)
H(13)	0.165(1)	0.573(4)	0.281(1)	3.3(5)
H(14)	-0.045(1)	-0.106(4)	0.154(2)	4.7(6)
H(15)	-0.152(2)	-0.254(5)	0.077(2)	6.0(7)
H(16)	-0.230(2)	-0.076(5)	-0.032(2)	6.5(7)
H(17)	-0.199(2)	0.241(5)	-0.058(2)	5.1(7)
H(18)	-0.092(1)	0.393(4)	0.021(1)	3.3(5)

$$B_{eq} = \frac{8}{3}\pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}aa^*bb^* \cos \gamma + 2U_{13}aa^*cc^* \cos \beta + 2U_{23}bb^*cc^* \cos \alpha)$$

Table 2. Anisotropic Displacement Parameters

atom	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
C(1)	0.032(1)	0.031(1)	0.030(1)	0.0005(10)	0.0102(9)	0.0006(10)
C(2)	0.034(1)	0.040(1)	0.032(1)	-0.007(1)	0.0095(9)	-0.005(1)
C(3)	0.039(1)	0.058(2)	0.031(1)	-0.013(1)	0.010(1)	-0.004(1)
C(4)	0.032(1)	0.056(2)	0.058(2)	0.000(1)	0.007(1)	0.010(2)
C(5)	0.035(1)	0.048(2)	0.062(2)	0.006(1)	0.018(1)	-0.004(1)
C(6)	0.045(2)	0.060(2)	0.058(2)	-0.014(1)	0.027(1)	-0.009(2)
C(7)	0.051(2)	0.037(2)	0.050(2)	-0.009(1)	0.020(1)	0.004(1)
C(8)	0.046(2)	0.039(2)	0.040(1)	0.000(1)	0.013(1)	0.006(1)
C(9)	0.040(1)	0.035(1)	0.054(2)	0.003(1)	0.017(1)	-0.005(1)
C(10)	0.040(1)	0.048(2)	0.051(2)	-0.012(1)	0.015(1)	-0.016(1)
C(11)	0.035(1)	0.041(1)	0.031(1)	0.001(1)	0.0108(10)	-0.005(1)
C(12)	0.041(1)	0.061(2)	0.037(1)	0.004(1)	0.012(1)	0.006(1)
C(13)	0.039(2)	0.107(3)	0.038(1)	0.003(2)	0.006(1)	0.002(2)
C(14)	0.042(2)	0.108(3)	0.053(2)	-0.022(2)	0.010(1)	-0.027(2)
C(15)	0.054(2)	0.058(2)	0.074(2)	-0.020(2)	0.021(2)	-0.022(2)
C(16)	0.045(2)	0.043(2)	0.049(2)	-0.006(1)	0.010(1)	-0.008(1)

The general temperature factor expression:

$$\exp(-2\pi^2(a^2U_{11}h^2 + b^2U_{22}k^2 + c^2U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$$

Table 3. Bond Lengths(Å)

atom	atom	distance	atom	atom	distance
C(1)	C(2)	1.548(3)	C(1)	C(8)	1.547(3)
C(1)	C(9)	1.557(3)	C(1)	C(11)	1.529(3)
C(2)	C(2)	1.351(4)	C(2)	C(3)	1.538(3)
C(3)	C(4)	1.534(4)	C(3)	C(10)	1.554(4)
C(3)	H(1)	0.92(2)	C(4)	C(5)	1.512(4)
C(4)	H(2)	0.98(3)	C(4)	H(3)	1.02(3)
C(5)	C(6)	1.516(4)	C(5)	C(9)	1.530(4)
C(5)	H(4)	1.04(3)	C(6)	C(7)	1.518(4)
C(6)	H(5)	1.01(3)	C(6)	H(6)	1.00(3)
C(7)	C(8)	1.531(4)	C(7)	C(10)	1.525(4)
C(7)	H(7)	0.98(3)	C(8)	H(8)	1.03(3)
C(8)	H(9)	1.00(2)	C(9)	H(10)	0.96(3)
C(9)	H(11)	1.05(3)	C(10)	H(12)	1.02(3)
C(10)	H(13)	0.96(2)	C(11)	C(12)	1.390(3)
C(11)	C(16)	1.398(4)	C(12)	C(13)	1.388(4)
C(12)	H(18)	0.98(3)	C(13)	C(14)	1.363(5)
C(13)	H(17)	0.94(3)	C(14)	C(15)	1.384(5)
C(14)	H(16)	0.94(3)	C(15)	C(16)	1.381(4)
C(15)	H(15)	0.97(3)	C(16)	H(14)	0.97(3)

Table 4. Bond Angles(°)

atom	atom	atom	angle	atom	atom	atom	angle
C(2)	C(1)	C(8)	108.4(2)	C(2)	C(1)	C(9)	108.4(2)
C(2)	C(1)	C(11)	116.0(2)	C(8)	C(1)	C(9)	106.5(2)
C(8)	C(1)	C(11)	112.6(2)	C(9)	C(1)	C(11)	104.4(2)
C(1)	C(2)	C(2)	127.1(3)	C(1)	C(2)	C(3)	109.2(2)
C(2)	C(2)	C(3)	123.0(3)	C(2)	C(3)	C(4)	114.0(2)
C(2)	C(3)	C(10)	106.4(2)	C(2)	C(3)	H(1)	112(1)
C(4)	C(3)	C(10)	108.4(2)	C(4)	C(3)	H(1)	109(1)
C(10)	C(3)	H(1)	105(1)	C(3)	C(4)	C(5)	109.5(2)
C(3)	C(4)	H(2)	108(1)	C(3)	C(4)	H(3)	108(1)
C(5)	C(4)	H(2)	111(1)	C(5)	C(4)	H(3)	111(1)
H(2)	C(4)	H(3)	106(2)	C(4)	C(5)	C(6)	109.9(3)
C(4)	C(5)	C(9)	107.9(2)	C(4)	C(5)	H(4)	109(1)
C(6)	C(5)	C(9)	110.2(2)	C(6)	C(5)	H(4)	110(1)
C(9)	C(5)	H(4)	109(1)	C(5)	C(6)	C(7)	109.6(2)
C(5)	C(6)	H(5)	110(1)	C(5)	C(6)	H(6)	111(1)
C(7)	C(6)	H(5)	111(1)	C(7)	C(6)	H(6)	110(1)
H(5)	C(6)	H(6)	104(2)	C(6)	C(7)	C(8)	110.1(2)
C(6)	C(7)	C(10)	109.7(2)	C(6)	C(7)	H(7)	111(1)
C(8)	C(7)	C(10)	109.0(2)	C(8)	C(7)	H(7)	108(1)
C(10)	C(7)	H(7)	108(1)	C(1)	C(8)	C(7)	110.7(2)
C(1)	C(8)	H(8)	109(1)	C(1)	C(8)	H(9)	109(1)
C(7)	C(8)	H(8)	110(1)	C(7)	C(8)	H(9)	108(1)
H(8)	C(8)	H(9)	108(1)	C(1)	C(9)	C(5)	112.6(2)
C(1)	C(9)	H(10)	108(1)	C(1)	C(9)	H(11)	107(1)

Table 4. Bond Angles(°) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
C(5)	C(9)	H(10)	108(1)	C(5)	C(9)	H(11)	111(1)
H(10)	C(9)	H(11)	107(2)	C(3)	C(10)	C(7)	109.7(2)
C(3)	C(10)	H(12)	109(1)	C(3)	C(10)	H(13)	108(1)
C(7)	C(10)	H(12)	110(1)	C(7)	C(10)	H(13)	111(1)
H(12)	C(10)	H(13)	108(2)	C(1)	C(11)	C(12)	123.1(2)
C(1)	C(11)	C(16)	119.5(2)	C(12)	C(11)	C(16)	117.3(3)
C(11)	C(12)	C(13)	120.9(3)	C(11)	C(12)	H(18)	118(1)
C(13)	C(12)	H(18)	120(1)	C(12)	C(13)	C(14)	120.7(3)
C(12)	C(13)	H(17)	118(1)	C(14)	C(13)	H(17)	120(1)
C(13)	C(14)	C(15)	119.7(3)	C(13)	C(14)	H(16)	119(2)
C(15)	C(14)	H(16)	120(2)	C(14)	C(15)	C(16)	119.8(3)
C(14)	C(15)	H(15)	122(1)	C(16)	C(15)	H(15)	118(1)
C(11)	C(16)	C(15)	121.5(3)	C(11)	C(16)	H(14)	118(1)
C(15)	C(16)	H(14)	119(1)				

Table 5. Torsion Angles(°)

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
C(1)	C(2)	C(2)	C(1)	167.2(3)	C(1)	C(2)	C(2)	C(3)	-23.2(2)
C(1)	C(2)	C(3)	C(4)	-55.5(3)	C(1)	C(2)	C(3)	C(10)	63.9(3)
C(1)	C(8)	C(7)	C(6)	62.0(3)	C(1)	C(8)	C(7)	C(10)	-58.4(3)
C(1)	C(9)	C(5)	C(4)	62.0(3)	C(1)	C(9)	C(5)	C(6)	-57.9(3)
C(1)	C(11)	C(12)	C(13)	178.1(3)	C(1)	C(11)	C(16)	C(15)	-177.5(3)
C(2)	C(1)	C(8)	C(7)	58.2(3)	C(2)	C(1)	C(9)	C(5)	-59.6(3)
C(2)	C(1)	C(11)	C(12)	123.2(3)	C(2)	C(1)	C(11)	C(16)	-60.3(3)
C(2)	C(2)	C(1)	C(8)	109.1(2)	C(2)	C(2)	C(1)	C(9)	-135.7(2)
C(2)	C(2)	C(1)	C(11)	-18.7(3)	C(2)	C(2)	C(3)	C(4)	133.3(2)
C(2)	C(2)	C(3)	C(10)	-107.3(2)	C(2)	C(3)	C(4)	C(5)	58.3(3)
C(2)	C(3)	C(10)	C(7)	-64.0(3)	C(3)	C(2)	C(1)	C(8)	-61.6(3)
C(3)	C(2)	C(1)	C(9)	53.6(3)	C(3)	C(2)	C(1)	C(11)	170.6(2)
C(3)	C(2)	C(2)	C(3)	146.3(4)	C(3)	C(4)	C(5)	C(6)	61.5(3)
C(3)	C(4)	C(5)	C(9)	-58.7(3)	C(3)	C(10)	C(7)	C(6)	-59.2(3)
C(3)	C(10)	C(7)	C(8)	61.5(3)	C(4)	C(3)	C(10)	C(7)	58.9(3)
C(4)	C(5)	C(6)	C(7)	-61.0(3)	C(5)	C(4)	C(3)	C(10)	-59.9(3)
C(5)	C(6)	C(7)	C(8)	-60.2(3)	C(5)	C(6)	C(7)	C(10)	59.8(3)
C(5)	C(9)	C(1)	C(8)	56.9(3)	C(5)	C(9)	C(1)	C(11)	176.1(2)
C(7)	C(6)	C(5)	C(9)	57.7(3)	C(7)	C(8)	C(1)	C(9)	-58.3(3)
C(7)	C(8)	C(1)	C(11)	-172.1(2)	C(8)	C(1)	C(11)	C(12)	-2.5(3)
C(8)	C(1)	C(11)	C(16)	174.0(2)	C(9)	C(1)	C(11)	C(12)	-117.6(3)
C(9)	C(1)	C(11)	C(16)	58.9(3)	C(11)	C(12)	C(13)	C(14)	-1.3(5)
C(11)	C(16)	C(15)	C(14)	-0.2(5)	C(12)	C(11)	C(16)	C(15)	-0.8(4)
C(12)	C(13)	C(14)	C(15)	0.2(5)	C(13)	C(12)	C(11)	C(16)	1.6(4)

Table 5. Torsion Angles(°) (continued)

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
C(13)	C(14)	C(15)	C(16)	0.6(5)					

Table 6. Non-bonded Contacts out to 3.60 Å

atom	atom	distance	ADC	atom	atom	distance	ADC
C(4)	H(6)	2.92(3)	54506	C(4)	H(17)	3.47(3)	8
C(5)	H(6)	3.40(3)	54506	C(5)	H(13)	3.53(3)	54506
C(5)	H(7)	3.55(3)	54501	C(6)	H(3)	3.34(3)	6
C(6)	H(17)	3.39(3)	56503	C(6)	H(18)	3.44(2)	56503
C(6)	H(16)	3.57(3)	3	C(6)	H(2)	3.59(3)	6
C(7)	H(10)	3.16(3)	56501	C(7)	H(18)	3.37(2)	56503
C(7)	H(17)	3.50(3)	56503	C(8)	H(9)	3.20(3)	56503
C(8)	H(18)	3.35(2)	56503	C(8)	H(14)	3.53(3)	56501
C(8)	H(10)	3.60(3)	56501	C(9)	H(7)	3.12(3)	54501
C(10)	H(15)	3.37(3)	56502	C(10)	H(10)	3.37(3)	56501
C(10)	H(2)	3.56(3)	56501	C(11)	H(11)	3.55(3)	3
C(12)	H(7)	3.25(3)	56503	C(12)	H(11)	3.31(3)	3
C(12)	H(9)	3.37(2)	56503	C(12)	H(5)	3.43(3)	56503
C(13)	H(11)	3.17(3)	3	C(13)	H(7)	3.17(3)	56503
C(13)	H(4)	3.23(3)	3	C(13)	H(17)	3.41(3)	45507
C(13)	H(3)	3.46(3)	45408	C(13)	H(5)	3.55(3)	56503
C(14)	H(5)	3.07(3)	3	C(14)	H(4)	3.24(3)	3
C(14)	H(11)	3.26(3)	3	C(14)	H(16)	3.36(3)	44507
C(14)	H(15)	3.38(3)	44507	C(14)	H(17)	3.39(3)	45507
C(15)	H(16)	3.17(3)	44507	C(15)	H(5)	3.27(3)	3
C(15)	H(8)	3.33(3)	54501	C(15)	H(11)	3.47(3)	3
C(15)	H(12)	3.54(3)	54502	C(15)	H(18)	3.55(3)	54501
C(16)	H(8)	3.10(3)	54501	C(16)	H(12)	3.37(3)	54502
H(1)	H(15)	3.18(4)	56502	H(1)	H(17)	3.57(4)	8

Table 6. Non-bonded Contacts out to 3.60 Å (continued)

atom	atom	distance	ADC	atom	atom	distance	ADC
H(1)	H(16)	3.60(4)	8	H(2)	H(6)	2.63(4)	54506
H(2)	H(12)	3.12(4)	54501	H(2)	H(13)	3.18(4)	54501
H(2)	H(17)	3.57(4)	8	H(3)	H(6)	2.52(4)	54506
H(3)	H(17)	2.60(4)	8	H(3)	H(4)	2.97(4)	6
H(3)	H(7)	3.24(4)	54506	H(3)	H(13)	3.45(4)	54506
H(3)	H(5)	3.56(4)	54506	H(4)	H(13)	2.77(4)	54506
H(4)	H(6)	2.85(4)	54506	H(4)	H(17)	3.05(4)	3
H(4)	H(16)	3.06(4)	3	H(4)	H(7)	3.06(4)	54501
H(5)	H(18)	2.71(4)	56503	H(5)	H(16)	2.87(4)	3
H(5)	H(17)	2.97(4)	56503	H(5)	H(15)	3.25(4)	3
H(6)	H(17)	3.21(4)	56503	H(6)	H(13)	3.22(4)	54506
H(7)	H(10)	2.42(4)	56501	H(7)	H(17)	2.79(4)	56503
H(7)	H(18)	2.96(3)	56503	H(7)	H(11)	3.17(4)	56501
H(8)	H(14)	2.57(4)	56501	H(8)	H(15)	3.02(4)	56501
H(8)	H(10)	3.10(4)	56501	H(8)	H(9)	3.27(4)	56503
H(9)	H(9)	2.32(5)	56503	H(9)	H(18)	2.59(3)	56503
H(10)	H(12)	2.87(4)	54501	H(10)	H(13)	3.58(4)	54501
H(11)	H(11)	3.30(5)	3	H(11)	H(17)	3.56(4)	3
H(12)	H(14)	2.69(4)	56502	H(12)	H(15)	3.00(4)	56502
H(12)	H(14)	3.11(4)	56501	H(13)	H(15)	2.97(4)	56502
H(13)	H(16)	3.34(4)	8	H(15)	H(16)	2.47(4)	44507
H(15)	H(18)	2.99(4)	54501	H(16)	H(16)	2.87(6)	44507
H(16)	H(17)	3.38(4)	45507	H(17)	H(17)	3.40(5)	45507

The ADC (atom designator code) specifies the position of an atom in a crystal. The 5-digit number shown in the table is a composite of three one-digit numbers and one two-digit number: TA (first digit) + TB (second digit) + TC (third digit) + SN (last two digits). TA, TB and TC are the crystal lattice translation digits along cell edges a, b and c. A translation digit of 5 indicates the origin unit cell. If TA = 4, this indicates a translation of one unit cell length along the a-axis in the negative direction. Each translation digit can range in value from 1 to 9 and thus ± 4 lattice translations from the origin (TA=5, TB=5, TC=5) can be represented.

The SN, or symmetry operator number, refers to the number of the symmetry operator used to generate the coordinates of the target atom. A list of symmetry operators relevant to this structure are given below.

For a given intermolecular contact, the first atom (origin atom) is located in the origin unit cell and its position can be generated using the identity operator (SN=1). Thus, the ADC for an origin atom is always 55501. The position of the second atom (target atom) can be generated using the ADC and the coordinates of the atom in the parameter table. For example, an ADC of 47502 refers to the target atom moved through symmetry operator two, then translated -1 cell translations along the a axis, +2 cell translations along the b axis, and 0 cell translations along the c axis.

An ADC of 1 indicates an intermolecular contact between two fragments (eg. cation and anion) that reside in the same asymmetric unit.

Symmetry Operators:

(1)	X,	Y,	Z	(2)	-X,	Y,	1/2-Z
(3)	-X,	-Y,	-Z	(4)	X,	-Y,	1/2+Z
(5)	1/2+X,	1/2+Y,	Z	(6)	1/2-X,	1/2+Y,	1/2-Z
(7)	1/2-X,	1/2-Y,	-Z	(8)	1/2+X,	1/2-Y,	1/2+Z

X-ray Structure Report

for 7

Mon Dec 11 2000

Experimental

Data Collection

A colorless prismatic crystal of $C_{32}H_{36}$ having approximate dimensions of 0.40 x 0.30 x 0.30 mm was mounted on a glass fiber. All measurements were made on a Rigaku AFC7R diffractometer with graphite monochromated Mo-K α radiation and a rotating anode generator.

Cell constants and an orientation matrix for data collection, obtained from a least-squares refinement using the setting angles of 25 carefully centered reflections in the range $28.84 < 2\theta < 30.00^\circ$ corresponded to a primitive monoclinic cell with dimensions:

$$\begin{aligned} a &= 10.270(7) \text{ \AA} \\ b &= 22.193(7) \text{ \AA} \quad \beta = 112.01(5)^\circ \\ c &= 10.763(8) \text{ \AA} \\ V &= 2274(2) \text{ \AA}^3 \end{aligned}$$

For $Z = 4$ and F.W. = 420.64, the calculated density is 1.23 g/cm³. The systematic absences of:

$$\begin{aligned} h0l: h+l &\neq 2n \\ 0k0: k &\neq 2n \end{aligned}$$

uniquely determine the space group to be:

$$P2_1/n \text{ (\#14)}$$

The data were collected at a temperature of $23 \pm 1^\circ\text{C}$ using the ω - 2θ scan technique to a maximum 2θ value of 55.0° . Omega scans of several intense reflections, made prior to data collection, had an average width at half-height of 0.22° with a take-off angle of 6.0° . Scans of $(1.31 + 0.30 \tan \theta)^\circ$ were made at a speed of $8.0^\circ/\text{min}$ (in omega). The weak reflections ($I < 10.0\sigma(I)$) were rescanned (maximum of 1 scans) and the counts were accumulated to ensure good counting statistics. Stationary background counts were recorded on each side of the reflection. The ratio of peak counting time to background counting time was 2:1. The diameter of the incident beam collimator was 1.0 mm and the crystal to detector distance was 235 mm. The computer-controlled slits were set to 3.0 mm (horizontal) and 3.5 mm (vertical).

Data Reduction

Of the 5495 reflections which were collected, 5219 were unique ($R_{\text{int}} = 0.080$). The intensities of three representative reflection were measured after every 150 reflections. No decay correction was applied.

The linear absorption coefficient, μ , for Mo-K α radiation is 0.7 cm^{-1} . An empirical absorption correction based on azimuthal scans of several reflections was applied which resulted in transmission factors ranging from 0.65 to 0.91. The data were corrected for Lorentz and polarization effects. A correction for secondary extinction was applied (coefficient = 1.11606×10^{-6}).

Structure Solution and Refinement

The structure was solved by direct methods¹ and expanded using Fourier techniques². The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined isotropically. The final cycle of full-matrix least-squares refinement³ was based on 1873 observed reflections ($I > 3.00\sigma(I)$) and 434 variable parameters and converged (largest parameter shift was 0.01 times its esd) with unweighted and weighted agreement factors of:

$$R = \Sigma ||Fo| - |Fc|| / \Sigma |Fo| = 0.049$$

$$R_w = \sqrt{\Sigma w(|Fo| - |Fc|)^2 / \Sigma wFo^2} = 0.049$$

The standard deviation of an observation of unit weight⁴ was 1.77. The weighting scheme was based on counting statistics and included a factor ($p = 0.002$) to downweight the intense reflections. Plots of $\Sigma w(|Fo| - |Fc|)^2$ versus $|Fo|$, reflection order in data collection, $\sin \theta/\lambda$ and various classes of indices showed no unusual trends. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.14 and -0.14 $e^-/\text{\AA}^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber⁵. Anomalous dispersion effects were included in Fcalc⁶; the values for $\Delta f'$ and $\Delta f''$ were those of Creagh and McAuley⁷. The values for the mass attenuation coefficients are those of Creagh and Hubbel⁸. All calculations were performed using the teXsan⁹ crystallographic software package of Molecular Structure Corporation.

References

(1) **SHELXS-97**: Sheldrick, G.M. (1997). Program for the Solution of Crystal Structures. University of Goettingen, Germany.

(2) **DIRDIF94**: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., de Gelder, R., Israel, R. and Smits, J.M.M. (1994). The DIRDIF-94 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

(3) Least-Squares:

Function minimized: $\Sigma w(|Fo| - |Fc|)^2$

where $w = \frac{1}{\sigma^2(Fo)} = [\sigma_c^2(Fo) + \frac{p^2}{4}Fo^2]^{-1}$

$\sigma_c(Fo)$ = e.s.d. based on counting statistics

p = p-factor

(4) Standard deviation of an observation of unit weight:

$$\sqrt{\Sigma w(|Fo| - |Fc|)^2 / (No - Nv)}$$

where: No = number of observations

Nv = number of variables

(5) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).

(6) Ibers, J. A. & Hamilton, W. C.; Acta Crystallogr., 17, 781 (1964).

(7) Creagh, D. C. & McAuley, W.J. ; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).

(8) Creagh, D. C. & Hubbell, J.H.; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200-206 (1992).

(9) teXsan: Crystal Structure Analysis Package, Molecular Structure Corporation (1985 & 1992).

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	C ₃₂ H ₃₆
Formula Weight	420.64
Crystal Color, Habit	colorless, prismatic
Crystal Dimensions	0.40 X 0.30 X 0.30 mm
Crystal System	monoclinic
Lattice Type	Primitive
No. of Reflections Used for Unit Cell Determination (2 θ range)	25 (28.8 - 30.0°)
Omega Scan Peak Width at Half-height	0.22°
Lattice Parameters	$a = 10.270(7) \text{ \AA}$ $b = 22.193(7) \text{ \AA}$ $c = 10.763(8) \text{ \AA}$ $\beta = 112.01(5)^\circ$
	$V = 2274(2) \text{ \AA}^3$
Space Group	P2 ₁ /n (#14)
Z value	4
D _{calc}	1.228 g/cm ³
F ₀₀₀	912.00
$\mu(\text{MoK}\alpha)$	0.69 cm ⁻¹

B. Intensity Measurements

Diffractometer	Rigaku AFC7R
Radiation	MoK α ($\lambda = 0.71069 \text{ \AA}$) graphite monochromated