Crystal structure of 1,6:3,4-bis(4,5-benzo-3,6-dimethoxy-1,2-xylylene)tetrahydro-3a,6a-diphenylimidazo[4,5-d]imidazole-2,5(1H,3H)-dione, C₄₄H₃₈N₄O₆

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Crystal structure of 1,6:3,4-bis(4,5-benzo-3,6-dimethoxy-1,2-xylylene) tetrahydro-3a,6a-diphenylimidazo[4,5-d]imidazole-2,5(1H,3H)-dione, $C_{44}H_{38}N_4O_6$

W. P. Bosman, P. T. Beurskens, G. Admiraal, R. P. Sijbesma and R. J. M. Nolte

Crystallography Laboratory and Dept. of Organic Chemistry, Tournooiveld, 6525 ED Nijmegen, The Netherlands

(Received April 26, 1991)

Source of material: Refluxing of 1,3,4,6-tetrakis(chloromethyl)tetrahydro-3a,6a-diphenylimidazo[4,5-d]imidazole-2,5(1H,3H)-dione and 1,4-dimethoxy-naphthalene in 1,2-dichloroethane using tin tetrachloride as a catalyst (see ref. 1).

Monoclinic, $P12_1/c1$ (no 14), $a = 17.242(2)$, $b = 11.208(2)$, $c = 19.536(2)$ Å, $\beta = 109.795(9)^\circ$, $V = 3552.3$ Å³, $Z = 4$, $R = 0.054$.

Table 1. Parameters used for the X-ray data collection

Diffraction type:	Enraf-Nonius CAD4	reflections:	
Wave length:	Mo K α radiation (0.7107 Å)	Criterion for unobserved reflections:	$F_o < 6\sigma(F_o)$
Crystal characteristics:	white needle, size 0.08 × 0.12 × 0.38 mm	Number of refined parameters:	353
Temperature of measurement:	293 K	Scan mode:	2 θ -scan
2 $\theta_{\max}$:	50°	μ :	0.8 g cm $^{-1}$ (see ref. 4)
Number of unique	6602	Structure solution program used:	ORIENT (see ref. 2) TRACOR (see ref. 3) SHELX

Table 2. Final atomic coordinates and displacement parameters (in Å 2)

Atom	x	y	z	U $_{11}$ /U $_{11}$	U $_{22}$	U $_{33}$	U $_{12}$	U $_{13}$	U $_{23}$
C(1)	0.9055(4)	0.3989(6)	0.5157(4)	0.027(4)	0.024(4)	0.027(5)	-0.007(4)	0.011(4)	-0.004(4)
C(2)	0.9519(4)	0.3465(6)	0.4784(4)	0.026(5)	0.030(5)	0.042(6)	-0.001(4)	0.009(4)	-0.001(4)
C(3)	0.9430(4)	0.3754(6)	0.4055(4)	0.044(5)	0.027(5)	0.027(5)	-0.013(4)	0.013(4)	-0.006(4)
C(4)	0.8854(4)	0.4642(6)	0.3702(4)	0.042(5)	0.029(5)	0.035(6)	-0.005(4)	0.020(4)	0.006(4)
C(5)	0.8372(4)	0.5167(6)	0.4092(4)	0.026(4)	0.032(5)	0.032(5)	0.002(4)	-0.000(4)	0.004(4)
C(6)	0.8457(4)	0.4849(6)	0.4787(4)	0.031(4)	0.022(4)	0.031(5)	-0.007(4)	0.013(4)	0.000(4)
O(1)	1.0153(3)	0.2676(4)	0.5141(2)	0.035(3)	0.040(3)	0.043(4)	0.008(3)	0.013(3)	0.002(3)
O(2)	0.7815(3)	0.6055(4)	0.3745(3)	0.048(3)	0.040(3)	0.039(4)	0.003(3)	0.005(3)	0.007(3)
C(7)	0.7939(4)	0.5454(6)	0.5173(4)	0.039(5)	0.036(5)	0.038(5)	0.011(5)	0.010(4)	0.005(5)
H(71)	0.7520(4)	0.5962(6)	0.4811(4)	0.03(2)					
H(72)	0.8308(4)	0.5970(6)	0.5567(4)	0.05(2)					
C(8)	0.9252(4)	0.3728(6)	0.5956(4)	0.023(4)	0.039(5)	0.028(5)	-0.002(4)	0.006(4)	-0.004(4)
H(81)	0.9467(4)	0.4478(6)	0.6232(4)	0.02(2)					
H(82)	0.9691(4)	0.3102(6)	0.6097(4)	0.03(2)					
N(1)	0.8569(3)	0.3305(5)	0.6165(3)	0.035(4)	0.021(4)	0.037(4)	1.000(3)	0.021(3)	-0.004(3)
C(9)	0.7920(4)	0.4085(6)	0.6206(4)	0.029(4)	0.031(4)	0.020(5)	0.003(4)	0.009(4)	-0.006(4)
N(2)	0.7502(3)	0.4638(5)	0.5502(3)	0.024(4)	0.031(4)	0.032(4)	-0.006(3)	0.006(3)	-0.001(3)
C(10)	0.6729(4)	0.4211(6)	0.5154(5)	0.032(5)	0.032(5)	0.041(6)	0.006(4)	0.010(5)	-0.000(5)
O(4)	0.6266(3)	0.4509(4)	0.4548(3)	0.045(3)	0.060(4)	0.043(4)	0.002(3)	0.005(3)	0.014(3)
N(3)	0.7607(3)	0.2030(5)	0.6193(3)	0.019(3)	0.026(4)	0.039(4)	-0.000(3)	0.018(3)	-0.004(3)
C(11)	0.7231(4)	0.3163(6)	0.6255(3)	0.023(4)	0.028(4)	0.016(4)	0.001(3)	0.000(3)	-0.002(4)
N(4)	0.6538(3)	0.3441(5)	0.5621(3)	0.034(4)	0.033(4)	0.027(4)	-0.003(3)	0.009(3)	0.003(3)
C(12)	0.8331(4)	0.2138(6)	0.6073(4)	0.041(5)	0.022(5)	0.029(5)	0.002(4)	0.018(4)	-0.001(4)
O(3)	0.8726(3)	0.1311(4)	0.5946(2)	0.041(3)	0.030(3)	0.035(3)	0.004(3)	0.014(3)	-0.003(3)
C(13)	0.7143(4)	0.0921(6)	0.6100(4)	0.029(4)	0.033(5)	0.035(5)	-0.007(4)	0.015(4)	0.000(4)
H(131)	0.7546(4)	0.0246(6)	0.6232(4)	0.09(3)					
H(132)	0.6827(4)	0.0940(6)	0.6445(4)	0.03(2)					
C(14)	0.5806(4)	0.2683(6)	0.5399(4)	0.031(5)	0.035(5)	0.046(6)	-0.002(4)	0.012(4)	-0.005(5)
H(141)	0.5683(4)	0.2463(6)	0.5847(4)	0.07(3)					
H(142)	0.5334	0.3149	0.5065	0.05756					
C(15)	0.5889(4)	0.1538(6)	0.5025(4)	0.024(4)	0.042(5)	0.023(5)	-0.012(4)	0.009(4)	-0.004(4)
C(16)	0.5299(4)	0.1222(6)	0.4385(4)	0.027(4)	0.042(5)	0.044(6)	0.005(4)	0.018(4)	0.004(5)
C(17)	0.5287(4)	0.0130(7)	0.4017(4)	0.031(5)	0.044(5)	0.031(5)	-0.006(4)	0.011(4)	-0.004(4)
C(18)	0.5934(4)	-0.0673(6)	0.4338(4)	0.031(5)	0.036(5)	0.040(6)	-0.008(4)	0.012(4)	-0.011(4)
C(19)	0.6562(4)	-0.0360(6)	0.5001(4)	0.035(5)	0.035(5)	0.036(5)	-0.002(4)	0.008(4)	-0.005(4)
C(20)	0.6531(4)	0.0679(6)	0.5347(4)	0.034(5)	0.028(5)	0.031(5)	-0.005(4)	0.009(4)	0.001(4)
O(5)	0.4616(3)	0.1972(4)	0.4068(3)	0.038(3)	0.057(4)	0.047(4)	0.001(3)	0.011(3)	0.003(3)
O(6)	0.7181(3)	-0.1183(4)	0.5315(3)	0.040(3)	0.039(3)	0.066(4)	0.008(3)	0.003(3)	-0.008(3)

Table 2. (Continued)

Atom	x	y	z	U _{iso} /U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
C(21)	0.8190(3)	0.4984(4)	0.6810(3)	0.036(5)	0.024(5)	0.040(5)	-0.008(4)	0.021(4)	-0.010(4)
C(22)	0.8815(3)	0.4692(4)	0.7457(2)	0.050(6)	0.047(6)	0.034(6)	-0.009(5)	0.014(5)	-0.001(5)
C(23)	0.9014(3)	0.5474(4)	0.8047(2)	0.073(7)	0.058(7)	0.038(7)	-0.020(6)	0.022(6)	-0.020(6)
C(24)	0.8588(3)	0.6549(4)	0.7989(2)	0.102(9)	0.056(7)	0.054(8)	-0.037(6)	0.042(5)	-0.037(5)
C(25)	0.7963(3)	0.6841(4)	0.7341(2)	0.086(8)	0.057(7)	0.066(8)	-0.014(6)	0.036(7)	-0.025(6)
C(26)	0.7765(3)	0.6059(4)	0.6751(2)	0.048(6)	0.044(6)	0.047(5)	-0.003(4)	0.015(4)	-0.018(4)
H(22)	0.9121(3)	0.3921(4)	0.7499(2)	0.010(3)					
H(23)	0.9461(3)	0.5264(4)	0.8512(2)	0.14(4)					
H(24)	0.8731(3)	0.7110(4)	0.8412(2)	0.09(3)					
H(25)	0.7658(3)	0.7612(4)	0.7299(2)	0.20(6)					
H(26)	0.7317(3)	0.6269(4)	0.6287(2)	0.04(2)					
C(27)	0.7036(3)	0.3265(4)	0.6957(2)	0.034(5)	0.022(5)	0.031(6)	0.000(5)	0.015(4)	-0.009(4)
C(28)	0.6440(3)	0.4072(4)	0.7006(2)	0.043(5)	0.039(5)	0.043(7)	0.012(8)	0.024(7)	0.002(5)
C(29)	0.6315(3)	0.4241(4)	0.7668(2)	0.058(6)	0.055(6)	0.053(7)	-0.001(5)	0.033(6)	-0.006(5)
C(30)	0.6785(3)	0.3604(4)	0.8282(2)	0.063(6)	0.054(6)	0.035(6)	-0.006(5)	0.035(5)	-0.015(5)
C(31)	0.7381(3)	0.2798(4)	0.8233(2)	0.048(6)	0.053(6)	0.033(6)	0.002(5)	0.017(5)	-0.006(5)
C(32)	0.7506(3)	0.2629(4)	0.7571(2)	0.047(5)	0.047(6)	0.026(5)	-0.005(4)	0.017(4)	-0.005(5)
H(28)	0.6103(3)	0.4528(4)	0.6566(2)	0.06(2)					
H(29)	0.5888(3)	0.4819(4)	0.7703(2)	0.13(4)					
H(30)	0.6695(3)	0.3726(4)	0.8757(2)	0.04(2)					
H(31)	0.7718(3)	0.2342(4)	0.8673(2)	0.07(3)					
H(32)	0.7933(3)	0.2051(4)	0.7536(2)	0.11(3)					
C(33)	0.9902(5)	0.3212(7)	0.3680(4)	0.054(6)	0.043(5)	0.039(6)	-0.000(5)	0.024(5)	-0.002(5)
H(33)	1.0314(5)	0.2582(7)	0.3923(4)	0.06(3)					
C(34)	0.9798(5)	0.3539(7)	0.2986(5)	0.053(6)	0.054(6)	0.050(7)	-0.014(5)	0.029(5)	-0.018(5)
H(34)	1.0125	0.313	0.2718	0.06598					
C(35)	0.9245(5)	0.4443(7)	0.2639(5)	0.044(6)	0.064(6)	0.035(6)	-0.027(5)	0.015(5)	-0.013(5)
H(35)	0.9191(5)	0.4686(7)	0.2133(5)	0.07(3)					
C(36)	0.8780(4)	0.4997(7)	0.2983(4)	0.042(5)	0.046(5)	0.029(6)	-0.011(4)	0.007(5)	0.007(4)
H(36)	0.8388(4)	0.5645(7)	0.2732(4)	0.07(2)					
C(37)	0.5955(5)	-0.1801(7)	0.4006(5)	0.041(5)	0.046(6)	0.055(6)	-0.011(5)	0.019(5)	-0.014(5)
H(37)	0.6416(5)	-0.2373(7)	0.4231(4)	0.15(4)					
C(38)	0.5336(5)	-0.2095(8)	0.3372(5)	0.057(6)	0.052(6)	0.053(6)	-0.019(5)	0.029(5)	-0.016(5)
H(38)	0.5335(5)	-0.2891(8)	0.3140(4)	0.14(4)					
C(39)	0.4692(5)	-0.1270(8)	0.3044(5)	0.044(6)	0.064(6)	0.043(6)	-0.017(5)	0.012(5)	-0.010(5)
H(39)	0.4257(5)	-0.1478(8)	0.2572(5)	0.06(3)					
C(40)	0.4656(5)	-0.0205(7)	0.3362(4)	0.035(5)	0.053(6)	0.038(6)	-0.006(5)	0.015(4)	0.003(5)
H(40)	0.4185(5)	0.0350(7)	0.3131(4)	0.08(3)					
C(41)	0.9932(6)	0.1446(6)	0.5010(5)	0.074(7)	0.030(5)	0.061(8)	0.011(5)	0.025(6)	0.004(5)
H(411)	0.9640(6)	0.1380(6)	0.4473(5)	0.07(3)					
H(412)	0.9562(6)	0.1142(6)	0.5269(5)	0.14(4)					
H(413)	1.0441(6)	0.0943(6)	0.5147(5)	0.10(3)					
C(42)	0.7026(5)	0.5630(8)	0.3273(5)	0.035(5)	0.073(7)	0.045(6)	0.014(5)	-0.005(5)	0.015(6)
H(421)	0.6726(5)	0.6339(8)	0.2998(5)	0.08(3)					
H(422)	0.6682(5)	0.5252(8)	0.3536(5)	0.16(5)					
H(423)	0.7110(5)	0.5041(8)	0.2920(5)	0.07(3)					
C(43)	0.7854(5)	-0.110(1)	0.5032(7)	0.035(6)	0.076(8)	0.12(1)	0.000(6)	0.031(6)	-0.036(7)
H(431)	0.7718(5)	-0.137(1)	0.4517(7)	0.13(5)					
H(432)	0.8277(5)	-0.165(1)	0.5356(7)	0.14(5)					
H(433)	0.8088(5)	-0.028(1)	0.5082(7)	0.20(6)					
C(44)	0.4698(7)	0.2753(9)	0.3514(6)	0.074(7)	0.073(8)	0.060(7)	0.001(7)	0.014(7)	0.022(7)
H(441)	0.4142(7)	0.3096(9)	0.3246(6)	0.21(6)					
H(442)	0.4918(7)	0.2365(9)	0.3156(6)	0.11(4)					
H(443)	0.5077(7)	0.3413(9)	0.3766(6)	0.10(4)					

Further details of the structure determination (e.g. structure factors) have been deposited within the relevant database and can be accessed as Collection No. 320244 or ordered from the Fachinformationszentrum Karlsruhe, D-7514 Eggenstein-Leopoldshafen.

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