Novel Pd(II) and Pt(II)-Catalyzed Regio- and Stereoselective *trans*-Hydroarylation of Alkynes by Simple Arenes

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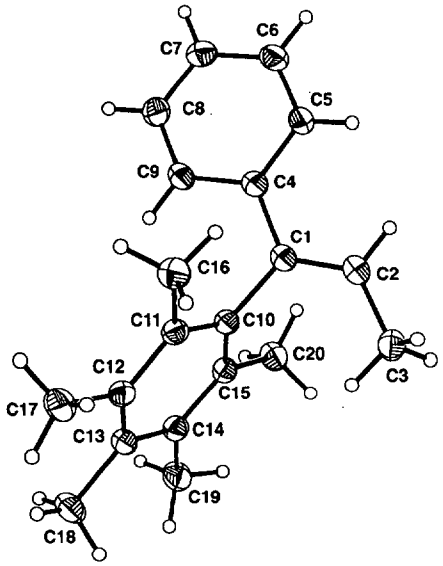
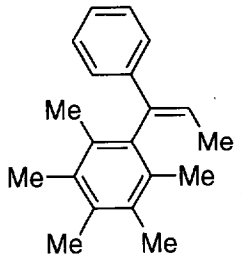
Supplementary Information

X-ray Diffraction Studies. Single crystal X-ray diffraction studies of **12** and **13** were performed on a Bruker SMART1000 CCD-based diffractometer (50 kV, 40 mA) with Mo *Ka* radiation. The data were collected as a series of o-scan frames, each with a width of 0.3° / frame at 121 K for **12** and 128 K for **13**. Crystal decay was monitored by repeating the 50 initial frames at the end data collection and analyzing the duplicate reflections. Data reduction was performed using SAINT software, which corrects for Lorentz and Polarization effects, and decay. The cell constants were calculated by the global refinement. The structure solved by direct methods using SHELXS-86 [1] and refined by full least-squares on f2 using SHELXL-97.[2] The positions of all hydrogen atoms were calculated geometrically and refined by the riding model.

There are two crystallographically independent molecules in the crystal of **12**, but they are almost identical except the angles around double bond. For clarity, one of the two independent molecules is shown in the main text. Crystallographical data for **12**: colorless needles, $C_{25}H_{26}$, FW = 326.46, orthorhombic (*Aba*2), a = 36.535(7) Å, b = 30.541(6) Å, c = 6.8017(13) Å, V = 7589(2) Å^3. Z = 16, μ (Mo$K\alpha$) = 0.064, R = 0.0813, wR_2 = 0.1783.





13

Crystallographical data for compound **13**: colorless prisms, $C_{20}H_{24}$, FW = 264.39, triclinic ($P\bar{1}$), a = 8.3713 (14) Å, b = 8.8839(15) Å, c = 10.7985(18) Å, α = 71.668(2)°, β = 83.231(3) °, γ = 85.820(3) °, V = 756.5(2) Å^3, Z = 2, μ(MoKa) = 0.065, R = 0.0531, wR_2 = 0.1637.

1. Sheldrick, G. M. *Acta Crystallogr., Sect. A* , **1990**, 46, 467

2. Sheldrick, G. M. *SHELXL-97, Program for Crystal Structure Refinement;* Universität Göttingen: Göttingen, 1997.

Table 1. Crystal data and structure refinement for **12**.

Identification code	**12**	
Empirical formula	C25 H26	
Formula weight	326.46	
Temperature	121(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	Aba2	
Unit cell dimensions	a = 36.535(7) Å	α= 90°.
	b = 30.541(6) Å	β= 90°.
	c = 6.8017(13) Å	γ = 90°.
Volume	7589(2) $Å^3$	
Z	16	
Density (calculated)	1.143 Mg/m^3	
Absorption coefficient	0.064 mm^{-1}	
F(000)	2816	
Crystal size	0.1 x 0.1 x 0.8 mm^3	
Theta range for data collection	1.33 to 27.90°.	
Index ranges	-41<=h<=47, -40<=k<=35, -8<=l<=8	
Reflections collected	25870	
Independent reflections	8126 [R(int) = 0.0882]	
Completeness to theta = 27.90°	94.4 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	8126 / 1 / 461	
Goodness-of-fit on F^2	1.039	
Final R indices [I>2sigma(I)]	R1 = 0.0813, wR2 = 0.1783	
R indices (all data)	R1 = 0.1297, wR2 = 0.2000	
Absolute structure parameter	-10(10)	
Largest diff. peak and hole	0.787 and -0.377 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **12**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	1120(1)	3173(3)	3148(8)	75(2)
C(2)	1397(1)	3084(3)	2306(8)	74(2)
C(3)	1709(1)	3317(2)	1438(6)	38(1)
C(4)	1774(1)	3769(1)	1435(6)	30(1)
C(5)	2081(1)	3940(1)	538(6)	37(1)
C(6)	2324(1)	3670(2)	-402(7)	47(1)
C(7)	2267(1)	3233(2)	-445(7)	50(1)
C(8)	1967(1)	3057(1)	448(7)	47(1)
C(9)	849(1)	2821(2)	3899(6)	41(1)
C(10)	669(1)	2920(1)	5626(7)	39(1)
C(11)	408(1)	2652(2)	6387(7)	53(1)
C(12)	317(1)	2264(2)	5430(10)	67(2)
C(13)	502(2)	2169(2)	3683(11)	73(2)
C(14)	758(1)	2442(2)	2974(9)	64(2)
C(15)	1039(1)	3660(1)	3592(6)	35(1)
C(16)	776(1)	3867(1)	2428(6)	30(1)
C(17)	642(1)	4273(1)	2980(6)	32(1)
C(18)	768(1)	4475(1)	4723(6)	30(1)
C(19)	1042(1)	4270(1)	5843(6)	28(1)
C(20)	1179(1)	3869(1)	5281(6)	32(1)
C(21)	632(1)	3646(1)	572(6)	41(1)
C(22)	356(1)	4504(2)	1735(7)	46(1)
C(23)	609(1)	4902(1)	5415(7)	45(1)
C(24)	1187(1)	4497(1)	7671(6)	37(1)
C(25)	1475(2)	3644(2)	6476(7)	54(1)
C(26)	1160(1)	1340(1)	9598(5)	27(1)
C(27)	1251(1)	1712(1)	8669(6)	28(1)
C(28)	1602(1)	1866(1)	7886(6)	27(1)
C(29)	1938(1)	1646(1)	8033(6)	31(1)
C(30)	2251(1)	1814(1)	7181(6)	31(1)
C(31)	2242(1)	2206(1)	6161(6)	36(1)
C(32)	1916(1)	2429(1)	6001(6)	37(1)

C(33)	1605(1)	2263(1)	6838(6)	31(1)
C(34)	775(1)	1259(1)	10230(6)	28(1)
C(35)	478(1)	1413(1)	9149(7)	36(1)
C(36)	125(1)	1340(2)	9745(7)	46(1)
C(37)	53(1)	1104(2)	11446(7)	44(1)
C(38)	345(1)	942(1)	12531(6)	37(1)
C(39)	701(1)	1015(1)	11937(6)	32(1)
C(40)	1431(1)	992(1)	10120(5)	25(1)
C(41)	1648(1)	1030(1)	11820(6)	27(1)
C(42)	1901(1)	701(1)	12276(6)	29(1)
C(43)	1926(1)	325(1)	11109(6)	33(1)
C(44)	1706(1)	285(1)	9414(6)	31(1)
C(45)	1460(1)	620(1)	8926(6)	26(1)
C(46)	1611(1)	1432(1)	13089(6)	35(1)
C(47)	2149(1)	754(2)	14058(7)	44(1)
C(48)	2181(1)	-41(1)	11688(7)	47(1)
C(49)	1733(1)	-114(1)	8116(7)	45(1)
C(50)	1226(1)	581(1)	7085(6)	38(1)

Table 3. Bond lengths [Å] and angles [°] for **12**.

C(1)-C(2)	1.192(8)	C(31)-C(32)	1.376(6)
C(1)-C(15)	1.545(8)	C(32)-C(33)	1.369(6)
C(1)-C(9)	1.551(7)	C(34)-C(35)	1.392(5)
C(2)-C(3)	1.470(7)	C(34)-C(39)	1.406(5)
C(3)-C(4)	1.401(6)	C(35)-C(36)	1.371(6)
C(3)-C(8)	1.404(6)	C(36)-C(37)	1.387(7)
C(4)-C(5)	1.378(6)	C(37)-C(38)	1.388(6)
C(5)-C(6)	1.370(6)	C(38)-C(39)	1.380(6)
C(6)-C(7)	1.350(7)	C(40)-C(45)	1.401(5)
C(7)-C(8)	1.363(7)	C(40)-C(41)	1.409(5)
C(9)-C(14)	1.357(7)	C(41)-C(42)	1.398(5)
C(9)-C(10)	1.380(6)	C(41)-C(46)	1.505(5)
C(10)-C(11)	1.357(6)	C(42)-C(43)	1.400(6)
C(11)-C(12)	1.392(8)	C(42)-C(47)	1.522(6)
C(12)-C(13)	1.399(9)	C(43)-C(44)	1.411(6)
C(13)-C(14)	1.344(8)	C(43)-C(48)	1.506(6)
C(15)-C(16)	1.397(6)	C(44)-C(45)	1.401(5)
C(15)-C(20)	1.411(6)	C(44)-C(49)	1.506(6)
C(16)-C(17)	1.385(6)	C(45)-C(50)	1.522(6)
C(16)-C(21)	1.526(5)		
C(17)-C(18)	1.412(6)	C(2)-C(1)-C(15)	118.5(6)
C(17)-C(22)	1.520(6)	C(2)-C(1)-C(9)	122.7(7)
C(18)-C(19)	1.406(6)	C(15)-C(1)-C(9)	118.7(5)
C(18)-C(23)	1.502(5)	C(1)-C(2)-C(3)	137.6(8)
C(19)-C(20)	1.377(5)	C(4)-C(3)-C(8)	116.3(4)
C(19)-C(24)	1.519(6)	C(4)-C(3)-C(2)	127.6(5)
C(20)-C(25)	1.517(6)	C(8)-C(3)-C(2)	116.1(5)
C(26)-C(27)	1.341(5)	C(5)-C(4)-C(3)	120.8(4)
C(26)-C(34)	1.491(5)	C(6)-C(5)-C(4)	120.4(4)
C(26)-C(40)	1.496(5)	C(7)-C(6)-C(5)	120.3(4)
C(27)-C(28)	1.468(5)	C(6)-C(7)-C(8)	120.3(4)
C(28)-C(29)	1.403(5)	C(7)-C(8)-C(3)	122.0(4)
C(28)-C(33)	1.407(5)	C(14)-C(9)-C(10)	117.8(4)
C(29)-C(30)	1.380(5)	C(14)-C(9)-C(1)	126.5(5)
C(30)-C(31)	1.384(6)	C(10)-C(9)-C(1)	115.7(4)

C(11)-C(10)-C(9)	121.8(4)	C(29)-C(30)-C(31)	120.7(4)
C(10)-C(11)-C(12)	120.2(5)	C(32)-C(31)-C(30)	119.3(4)
C(11)-C(12)-C(13)	117.3(5)	C(33)-C(32)-C(31)	120.1(4)
C(14)-C(13)-C(12)	120.9(5)	C(32)-C(33)-C(28)	122.4(4)
C(13)-C(14)-C(9)	122.1(5)	C(35)-C(34)-C(39)	117.8(4)
C(16)-C(15)-C(20)	120.3(3)	C(35)-C(34)-C(26)	121.8(4)
C(16)-C(15)-C(1)	117.2(4)	C(39)-C(34)-C(26)	120.5(3)
C(20)-C(15)-C(1)	121.7(4)	C(36)-C(35)-C(34)	121.5(4)
C(17)-C(16)-C(15)	119.7(4)	C(35)-C(36)-C(37)	120.5(4)
C(17)-C(16)-C(21)	119.9(4)	C(36)-C(37)-C(38)	119.1(4)
C(15)-C(16)-C(21)	120.4(3)	C(39)-C(38)-C(37)	120.6(4)
C(16)-C(17)-C(18)	120.3(4)	C(38)-C(39)-C(34)	120.5(4)
C(16)-C(17)-C(22)	120.5(4)	C(45)-C(40)-C(41)	119.9(3)
C(18)-C(17)-C(22)	119.2(4)	C(45)-C(40)-C(26)	119.4(3)
C(19)-C(18)-C(17)	119.4(3)	C(41)-C(40)-C(26)	120.7(3)
C(19)-C(18)-C(23)	119.4(4)	C(42)-C(41)-C(40)	119.7(3)
C(17)-C(18)-C(23)	121.1(4)	C(42)-C(41)-C(46)	121.2(4)
C(20)-C(19)-C(18)	120.3(4)	C(40)-C(41)-C(46)	119.1(3)
C(20)-C(19)-C(24)	120.4(4)	C(41)-C(42)-C(43)	120.5(4)
C(18)-C(19)-C(24)	119.3(3)	C(41)-C(42)-C(47)	119.6(4)
C(19)-C(20)-C(15)	119.8(4)	C(43)-C(42)-C(47)	119.9(4)
C(19)-C(20)-C(25)	120.9(4)	C(42)-C(43)-C(44)	119.8(4)
C(15)-C(20)-C(25)	119.2(3)	C(42)-C(43)-C(48)	120.0(4)
C(27)-C(26)-C(34)	120.7(3)	C(44)-C(43)-C(48)	120.1(4)
C(27)-C(26)-C(40)	123.3(3)	C(45)-C(44)-C(43)	119.7(3)
C(34)-C(26)-C(40)	116.0(3)	C(45)-C(44)-C(49)	119.6(4)
C(26)-C(27)-C(28)	131.3(4)	C(43)-C(44)-C(49)	120.8(4)
C(29)-C(28)-C(33)	116.3(3)	C(44)-C(45)-C(40)	120.3(4)
C(29)-C(28)-C(27)	125.8(3)	C(44)-C(45)-C(50)	119.9(4)
C(33)-C(28)-C(27)	117.8(3)	C(40)-C(45)-C(50)	119.8(3)
C(30)-C(29)-C(28)	121.1(4)		

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **12**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	38(3)	160(6)	26(3)	15(3)	-13(2)	-19(3)
C(2)	42(3)	152(6)	29(3)	33(3)	-15(3)	-29(3)
C(3)	40(2)	58(3)	15(2)	4(2)	-11(2)	-5(2)
C(4)	25(2)	42(2)	21(2)	-7(2)	-3(2)	6(2)
C(5)	45(2)	43(2)	24(2)	2(2)	-4(2)	-8(2)
C(6)	31(2)	89(4)	20(2)	11(2)	4(2)	0(2)
C(7)	49(3)	72(3)	28(2)	-8(2)	-3(2)	26(3)
C(8)	71(3)	35(2)	34(3)	-1(2)	-16(3)	12(2)
C(9)	26(2)	73(3)	24(2)	2(2)	-2(2)	-13(2)
C(10)	52(3)	33(2)	31(2)	-6(2)	-2(2)	-4(2)
C(11)	36(3)	86(4)	36(3)	16(3)	10(2)	6(3)
C(12)	52(3)	58(3)	90(5)	45(3)	-37(3)	-36(3)
C(13)	72(4)	49(3)	99(5)	-26(3)	-44(4)	6(3)
C(14)	39(3)	90(4)	63(4)	-44(3)	-8(3)	2(3)
C(15)	52(3)	24(2)	28(2)	-8(2)	-10(2)	9(2)
C(16)	39(2)	31(2)	20(2)	2(2)	-6(2)	-6(2)
C(17)	22(2)	41(2)	34(2)	8(2)	2(2)	-1(2)
C(18)	30(2)	31(2)	30(2)	-5(2)	10(2)	-1(2)
C(19)	34(2)	25(2)	25(2)	-4(2)	4(2)	-9(2)
C(20)	46(2)	32(2)	19(2)	-4(2)	-6(2)	5(2)
C(21)	52(3)	40(2)	28(2)	-3(2)	-15(2)	-2(2)
C(22)	35(2)	61(3)	43(3)	4(2)	-2(2)	17(2)
C(23)	48(3)	46(2)	41(3)	0(2)	6(2)	13(2)
C(24)	48(2)	34(2)	29(2)	-11(2)	7(2)	-9(2)
C(25)	75(3)	57(3)	29(2)	-12(2)	-23(2)	26(3)
C(26)	31(2)	32(2)	17(2)	-2(2)	-1(2)	-2(2)
C(27)	35(2)	30(2)	19(2)	-3(2)	-4(2)	6(2)
C(28)	30(2)	34(2)	17(2)	-6(2)	-1(2)	-7(2)
C(29)	28(2)	40(2)	24(2)	6(2)	-3(2)	-4(2)
C(30)	27(2)	41(2)	27(2)	-7(2)	-6(2)	-2(2)
C(31)	46(3)	38(2)	24(2)	-8(2)	9(2)	-15(2)
C(32)	51(3)	32(2)	27(2)	-3(2)	6(2)	-10(2)

C(33)	38(2)	29(2)	25(2)	-7(2)	-3(2)	3(2)
C(34)	30(2)	26(2)	26(2)	-3(2)	0(2)	-1(2)
C(35)	32(2)	44(2)	31(2)	0(2)	-3(2)	-1(2)
C(36)	26(2)	71(3)	41(3)	-7(2)	-4(2)	-4(2)
C(37)	27(2)	59(3)	45(3)	-25(2)	8(2)	-11(2)
C(38)	38(2)	40(2)	32(2)	-6(2)	11(2)	-2(2)
C(39)	39(2)	31(2)	26(2)	-4(2)	1(2)	-5(2)
C(40)	27(2)	25(2)	22(2)	2(2)	0(2)	-2(2)
C(41)	31(2)	35(2)	16(2)	2(2)	3(2)	-4(2)
C(42)	27(2)	36(2)	23(2)	8(2)	5(2)	-3(2)
C(43)	35(2)	29(2)	35(2)	11(2)	13(2)	-3(2)
C(44)	30(2)	28(2)	35(2)	-3(2)	16(2)	-6(2)
C(45)	26(2)	27(2)	25(2)	1(2)	8(2)	-11(2)
C(46)	36(2)	44(2)	24(2)	-6(2)	-5(2)	1(2)
C(47)	43(3)	54(3)	33(3)	12(2)	-6(2)	10(2)
C(48)	49(3)	46(3)	46(3)	15(2)	11(2)	12(2)
C(49)	46(3)	40(2)	47(3)	-7(2)	18(2)	1(2)
C(50)	37(2)	49(3)	28(2)	-13(2)	3(2)	-9(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for **12**.

	x	y	z	U(eq)
H(2A)	1421	2776	2154	89
H(4A)	1605	3960	2060	35
H(5A)	2124	4246	571	45
H(6A)	2534	3790	-1026	56
H(7A)	2436	3047	-1100	60
H(8A)	1931	2749	399	56
H(10A)	728	3183	6301	46
H(11A)	288	2730	7575	63
H(12A)	136	2072	5942	80
H(13A)	447	1907	2989	88
H(14A)	880	2368	1785	77
H(21A)	364	3625	643	61
H(21B)	736	3352	464	61
H(21C)	701	3819	-582	61
H(22A)	361	4386	396	69
H(22B)	408	4819	1699	69
H(22C)	113	4457	2308	69
H(23A)	554	4883	6823	67
H(23B)	383	4963	4688	67
H(23C)	785	5138	5184	67
H(24A)	1396	4333	8188	55
H(24B)	994	4510	8669	55
H(24C)	1264	4795	7335	55
H(25A)	1401	3634	7860	80
H(25B)	1704	3807	6352	80
H(25C)	1509	3345	5986	80
H(27A)	1053	1910	8485	34
H(29A)	1950	1377	8732	37
H(30A)	2475	1658	7296	38
H(31A)	2458	2319	5576	44
H(32A)	1907	2699	5309	44
H(33A)	1383	2422	6706	37

H(35A)	521	1572	7971	43
H(36A)	-73	1452	8987	55
H(37A)	-192	1055	11861	53
H(38A)	299	779	13695	44
H(39A)	898	899	12687	38
H(46A)	1607	1345	14476	52
H(46B)	1383	1584	12760	52
H(46C)	1820	1627	12857	52
H(47A)	2133	1055	14545	65
H(47B)	2402	688	13684	65
H(47C)	2071	551	15095	65
H(48A)	2173	-271	10687	71
H(48B)	2104	-161	12958	71
H(48C)	2431	73	11795	71
H(49A)	1942	-292	8527	67
H(49B)	1766	-22	6748	67
H(49C)	1508	-286	8230	67
H(50A)	1059	832	7007	57
H(50B)	1083	310	7140	57
H(50C)	1384	577	5921	57

Table 6. Torsion angles [°] for **12**.

C(15)-C(1)-C(2)-C(3)	-2.0(9)	C(21)-C(16)-C(17)-C(22)	-0.9(6)
C(9)-C(1)-C(2)-C(3)	-179.1(5)	C(16)-C(17)-C(18)-C(19)	3.1(5)
C(1)-C(2)-C(3)-C(4)	2.4(9)	C(22)-C(17)-C(18)-C(19)	-177.8(4)
C(1)-C(2)-C(3)-C(8)	-175.7(6)	C(16)-C(17)-C(18)-C(23)	-176.1(4)
C(8)-C(3)-C(4)-C(5)	-1.4(6)	C(22)-C(17)-C(18)-C(23)	3.1(6)
C(2)-C(3)-C(4)-C(5)	-179.4(4)	C(17)-C(18)-C(19)-C(20)	-2.2(6)
C(3)-C(4)-C(5)-C(6)	1.2(6)	C(23)-C(18)-C(19)-C(20)	177.0(4)
C(4)-C(5)-C(6)-C(7)	-0.5(7)	C(17)-C(18)-C(19)-C(24)	177.5(3)
C(5)-C(6)-C(7)-C(8)	-0.1(7)	C(23)-C(18)-C(19)-C(24)	-3.4(5)
C(6)-C(7)-C(8)-C(3)	-0.1(7)	C(18)-C(19)-C(20)-C(15)	-1.0(6)
C(4)-C(3)-C(8)-C(7)	0.9(6)	C(24)-C(19)-C(20)-C(15)	179.3(4)
C(2)-C(3)-C(8)-C(7)	179.1(4)	C(18)-C(19)-C(20)-C(25)	-179.9(4)
C(2)-C(1)-C(9)-C(14)	-38.0(8)	C(24)-C(19)-C(20)-C(25)	0.5(6)
C(15)-C(1)-C(9)-C(14)	145.0(5)	C(16)-C(15)-C(20)-C(19)	3.4(6)
C(2)-C(1)-C(9)-C(10)	146.0(5)	C(1)-C(15)-C(20)-C(19)	-166.6(4)
C(15)-C(1)-C(9)-C(10)	-31.0(6)	C(16)-C(15)-C(20)-C(25)	-177.7(4)
C(14)-C(9)-C(10)-C(11)	-0.3(7)	C(1)-C(15)-C(20)-C(25)	12.3(7)
C(1)-C(9)-C(10)-C(11)	176.1(4)	C(34)-C(26)-C(27)-C(28)	-179.1(4)
C(9)-C(10)-C(11)-C(12)	0.3(7)	C(40)-C(26)-C(27)-C(28)	2.8(6)
C(10)-C(11)-C(12)-C(13)	-0.2(7)	C(26)-C(27)-C(28)-C(29)	-2.8(7)
C(11)-C(12)-C(13)-C(14)	0.1(8)	C(26)-C(27)-C(28)-C(33)	175.2(4)
C(12)-C(13)-C(14)-C(9)	-0.1(8)	C(33)-C(28)-C(29)-C(30)	-0.3(6)
C(10)-C(9)-C(14)-C(13)	0.2(8)	C(27)-C(28)-C(29)-C(30)	177.7(4)
C(1)-C(9)-C(14)-C(13)	-175.8(5)	C(28)-C(29)-C(30)-C(31)	0.2(6)
C(2)-C(1)-C(15)-C(16)	104.5(6)	C(29)-C(30)-C(31)-C(32)	0.1(6)
C(9)-C(1)-C(15)-C(16)	-78.3(5)	C(30)-C(31)-C(32)-C(33)	-0.3(6)
C(2)-C(1)-C(15)-C(20)	-85.2(6)	C(31)-C(32)-C(33)-C(28)	0.3(6)
C(9)-C(1)-C(15)-C(20)	92.0(5)	C(29)-C(28)-C(33)-C(32)	0.0(6)
C(20)-C(15)-C(16)-C(17)	-2.5(6)	C(27)-C(28)-C(33)-C(32)	-178.1(4)
C(1)-C(15)-C(16)-C(17)	167.9(4)	C(27)-C(26)-C(34)-C(35)	33.4(5)
C(20)-C(15)-C(16)-C(21)	178.5(4)	C(40)-C(26)-C(34)-C(35)	-148.4(4)
C(1)-C(15)-C(16)-C(21)	-11.1(6)	C(27)-C(26)-C(34)-C(39)	-147.8(4)
C(15)-C(16)-C(17)-C(18)	-0.8(6)	C(40)-C(26)-C(34)-C(39)	30.4(5)
C(21)-C(16)-C(17)-C(18)	178.3(4)	C(39)-C(34)-C(35)-C(36)	1.7(6)
C(15)-C(16)-C(17)-C(22)	-179.9(4)	C(26)-C(34)-C(35)-C(36)	-179.5(4)

C(34)-C(35)-C(36)-C(37)	-0.8(7)	C(46)-C(41)-C(42)-C(47)	-1.4(5)
C(35)-C(36)-C(37)-C(38)	-0.3(7)	C(41)-C(42)-C(43)-C(44)	2.6(5)
C(36)-C(37)-C(38)-C(39)	0.3(6)	C(47)-C(42)-C(43)-C(44)	-177.5(3)
C(37)-C(38)-C(39)-C(34)	0.7(6)	C(41)-C(42)-C(43)-C(48)	-175.9(3)
C(35)-C(34)-C(39)-C(38)	-1.6(5)	C(47)-C(42)-C(43)-C(48)	3.9(5)
C(26)-C(34)-C(39)-C(38)	179.6(4)	C(42)-C(43)-C(44)-C(45)	-0.8(5)
C(27)-C(26)-C(40)-C(45)	-99.8(4)	C(48)-C(43)-C(44)-C(45)	177.8(4)
C(34)-C(26)-C(40)-C(45)	82.0(4)	C(42)-C(43)-C(44)-C(49)	178.9(4)
C(27)-C(26)-C(40)-C(41)	81.7(5)	C(48)-C(43)-C(44)-C(49)	-2.5(5)
C(34)-C(26)-C(40)-C(41)	-96.5(4)	C(43)-C(44)-C(45)-C(40)	-0.5(5)
C(45)-C(40)-C(41)-C(42)	1.8(5)	C(49)-C(44)-C(45)-C(40)	179.7(3)
C(26)-C(40)-C(41)-C(42)	-179.8(3)	C(43)-C(44)-C(45)-C(50)	179.2(3)
C(45)-C(40)-C(41)-C(46)	-179.7(3)	C(49)-C(44)-C(45)-C(50)	-0.5(5)
C(26)-C(40)-C(41)-C(46)	-1.3(5)	C(41)-C(40)-C(45)-C(44)	0.1(5)
C(40)-C(41)-C(42)-C(43)	-3.1(5)	C(26)-C(40)-C(45)-C(44)	-178.4(3)
C(46)-C(41)-C(42)-C(43)	178.4(3)	C(41)-C(40)-C(45)-C(50)	-179.7(3)
C(40)-C(41)-C(42)-C(47)	177.0(3)	C(26)-C(40)-C(45)-C(50)	1.9(5)

Symmetry transformations used to generate equivalent atoms:

Table 7. Crystal data and structure refinement for **13**.

Identification code	**13**	
Empirical formula	C20 H24	
Formula weight	264.39	
Temperature	128(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 8.3713(14) Å	α= 71.668(2)°.
	b = 8.8839(15) Å	β= 83.231(3)°.
	c = 10.7985(18) Å	γ = 85.820(3)°.
Volume	756.5(2) $Å^3$	
Z	2	
Density (calculated)	1.161 Mg/m^3	
Absorption coefficient	0.065 mm^{-1}	
F(000)	288	
Crystal size	0.6 x 0.6 x 0.6 mm^3	
Theta range for data collection	2.00 to 27.87°.	
Index ranges	-10<=h<=9, -11<=k<=11, -13<=l<=13	
Reflections collected	6217	
Independent reflections	3157 [R(int) = 0.0280]	
Completeness to theta = 27.87°	87.8 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3157 / 0 / 187	
Goodness-of-fit on F^2	1.265	
Final R indices [I>2sigma(I)]	R1 = 0.0531, wR2 = 0.1637	
R indices (all data)	R1 = 0.0664, wR2 = 0.1724	
Largest diff. peak and hole	0.316 and -0.282 $e.Å^{-3}$	

Table 8. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **13**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	1570(2)	3674(2)	2052(1)	21(1)
C(2)	1627(2)	4929(2)	962(1)	24(1)
C(3)	2750(2)	5121(2)	-250(1)	28(1)
C(4)	393(2)	3579(2)	3214(1)	22(1)
C(5)	-731(2)	4816(2)	3276(1)	26(1)
C(6)	-1827(2)	4677(2)	4357(2)	30(1)
C(7)	-1849(2)	3310(2)	5422(2)	29(1)
C(8)	-740(2)	2088(2)	5396(2)	29(1)
C(9)	364(2)	2223(2)	4309(1)	25(1)
C(10)	2702(2)	2266(2)	2120(1)	21(1)
C(11)	4118(2)	2107(2)	2730(1)	23(1)
C(12)	5182(2)	801(2)	2784(1)	25(1)
C(13)	4830(2)	-338(2)	2210(1)	25(1)
C(14)	3416(2)	-177(2)	1602(1)	24(1)
C(15)	2333(2)	1107(2)	1576(1)	22(1)
C(16)	4495(2)	3336(2)	3347(2)	28(1)
C(17)	6690(2)	654(2)	3465(2)	34(1)
C(18)	5982(2)	-1737(2)	2224(2)	35(1)
C(19)	3031(2)	-1394(2)	978(2)	33(1)
C(20)	769(2)	1245(2)	976(2)	27(1)

Table 9. Bond lengths [Å] and angles [°] for **13**.

C(1)-C(2)	1.341(2)	C(9)-C(4)-C(5)	116.93(13)
C(1)-C(4)	1.486(2)	C(9)-C(4)-C(1)	120.35(12)
C(1)-C(10)	1.5011(18)	C(5)-C(4)-C(1)	122.72(13)
C(2)-C(3)	1.489(2)	C(6)-C(5)-C(4)	121.26(13)
C(4)-C(9)	1.397(2)	C(5)-C(6)-C(7)	120.72(13)
C(4)-C(5)	1.4065(19)	C(8)-C(7)-C(6)	119.17(14)
C(5)-C(6)	1.376(2)	C(7)-C(8)-C(9)	120.28(14)
C(6)-C(7)	1.385(2)	C(8)-C(9)-C(4)	121.62(13)
C(7)-C(8)	1.381(2)	C(11)-C(10)-C(15)	120.14(12)
C(8)-C(9)	1.385(2)	C(11)-C(10)-C(1)	120.04(12)
C(10)-C(11)	1.400(2)	C(15)-C(10)-C(1)	119.82(12)
C(10)-C(15)	1.4034(19)	C(10)-C(11)-C(12)	120.09(13)
C(11)-C(12)	1.4012(19)	C(10)-C(11)-C(16)	120.13(12)
C(11)-C(16)	1.5145(19)	C(12)-C(11)-C(16)	119.77(13)
C(12)-C(13)	1.406(2)	C(11)-C(12)-C(13)	119.66(13)
C(12)-C(17)	1.512(2)	C(11)-C(12)-C(17)	118.75(14)
C(13)-C(14)	1.397(2)	C(13)-C(12)-C(17)	121.59(13)
C(13)-C(18)	1.5141(19)	C(14)-C(13)-C(12)	120.12(13)
C(14)-C(15)	1.3995(19)	C(14)-C(13)-C(18)	119.19(13)
C(14)-C(19)	1.513(2)	C(12)-C(13)-C(18)	120.69(14)
C(15)-C(20)	1.509(2)	C(13)-C(14)-C(15)	120.26(13)
		C(13)-C(14)-C(19)	120.47(13)
C(2)-C(1)-C(4)	122.93(13)	C(15)-C(14)-C(19)	119.28(13)
C(2)-C(1)-C(10)	119.96(13)	C(14)-C(15)-C(10)	119.69(13)
C(4)-C(1)-C(10)	117.09(12)	C(14)-C(15)-C(20)	120.42(12)
C(1)-C(2)-C(3)	126.64(13)	C(10)-C(15)-C(20)	119.89(12)

Symmetry transformations used to generate equivalent atoms:

Table 10. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **13**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	20(1)	21(1)	24(1)	-9(1)	-6(1)	0(1)
C(2)	24(1)	22(1)	29(1)	-8(1)	-5(1)	1(1)
C(3)	31(1)	26(1)	26(1)	-5(1)	-4(1)	-1(1)
C(4)	21(1)	22(1)	24(1)	-10(1)	-6(1)	0(1)
C(5)	29(1)	25(1)	25(1)	-8(1)	-6(1)	6(1)
C(6)	28(1)	32(1)	31(1)	-13(1)	-6(1)	9(1)
C(7)	27(1)	35(1)	27(1)	-13(1)	2(1)	-1(1)
C(8)	34(1)	25(1)	28(1)	-7(1)	-1(1)	-2(1)
C(9)	27(1)	21(1)	29(1)	-9(1)	-5(1)	2(1)
C(10)	21(1)	20(1)	20(1)	-5(1)	0(1)	0(1)
C(11)	21(1)	26(1)	21(1)	-7(1)	-1(1)	-1(1)
C(12)	21(1)	29(1)	22(1)	-5(1)	0(1)	1(1)
C(13)	26(1)	22(1)	24(1)	-5(1)	2(1)	4(1)
C(14)	29(1)	21(1)	22(1)	-6(1)	1(1)	0(1)
C(15)	25(1)	21(1)	20(1)	-6(1)	-1(1)	-1(1)
C(16)	25(1)	32(1)	30(1)	-14(1)	-5(1)	-1(1)
C(17)	26(1)	41(1)	36(1)	-12(1)	-9(1)	8(1)
C(18)	33(1)	28(1)	40(1)	-8(1)	-3(1)	9(1)
C(19)	44(1)	24(1)	35(1)	-12(1)	-7(1)	4(1)
C(20)	29(1)	26(1)	30(1)	-11(1)	-8(1)	1(1)

Table 11. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for **13**.

	x	y	z	U(eq)
H(2A)	874	5782	963	29
H(3A)	3380	6065	-419	42
H(3B)	2132	5242	-993	42
H(3C)	3478	4182	-133	42
H(5A)	-733	5766	2557	31
H(6A)	-2576	5529	4373	36
H(7A)	-2619	3213	6160	35
H(8A)	-735	1151	6126	35
H(9A)	1121	1373	4308	30
H(16A)	3626	4154	3238	42
H(16B)	5510	3825	2919	42
H(16C)	4592	2824	4284	42
H(17A)	7249	1656	3130	51
H(17B)	7399	-202	3296	51
H(17C)	6399	414	4411	51
H(18A)	6292	-1749	1323	52
H(18B)	5455	-2724	2730	52
H(18C)	6944	-1645	2627	52
H(19A)	2255	-925	336	50
H(19B)	2568	-2316	1656	50
H(19C)	4019	-1727	536	50
H(20A)	75	2086	1187	41
H(20B)	226	235	1328	41
H(20C)	988	1504	22	41

Table 12. Torsion angles [°] for **13**.

C(4)-C(1)-C(2)-C(3)	-179.45(13)	C(1)-C(10)-C(11)-C(16)	1.2(2)
C(10)-C(1)-C(2)-C(3)	-0.9(2)	C(10)-C(11)-C(12)-C(13)	0.7(2)
C(2)-C(1)-C(4)-C(9)	178.37(14)	C(16)-C(11)-C(12)-C(13)	-179.98(13)
C(10)-C(1)-C(4)-C(9)	-0.18(19)	C(10)-C(11)-C(12)-C(17)	-179.24(13)
C(2)-C(1)-C(4)-C(5)	-1.5(2)	C(16)-C(11)-C(12)-C(17)	0.1(2)
C(10)-C(1)-C(4)-C(5)	179.91(12)	C(11)-C(12)-C(13)-C(14)	-0.7(2)
C(9)-C(4)-C(5)-C(6)	-1.3(2)	C(17)-C(12)-C(13)-C(14)	179.19(14)
C(1)-C(4)-C(5)-C(6)	178.62(13)	C(11)-C(12)-C(13)-C(18)	178.41(13)
C(4)-C(5)-C(6)-C(7)	0.2(2)	C(17)-C(12)-C(13)-C(18)	-1.7(2)
C(5)-C(6)-C(7)-C(8)	0.9(2)	C(12)-C(13)-C(14)-C(15)	-0.8(2)
C(6)-C(7)-C(8)-C(9)	-1.0(2)	C(18)-C(13)-C(14)-C(15)	-179.95(13)
C(7)-C(8)-C(9)-C(4)	-0.2(2)	C(12)-C(13)-C(14)-C(19)	179.78(13)
C(5)-C(4)-C(9)-C(8)	1.3(2)	C(18)-C(13)-C(14)-C(19)	0.6(2)
C(1)-C(4)-C(9)-C(8)	-178.66(13)	C(13)-C(14)-C(15)-C(10)	2.4(2)
C(2)-C(1)-C(10)-C(11)	97.67(17)	C(19)-C(14)-C(15)-C(10)	-178.21(13)
C(4)-C(1)-C(10)-C(11)	-83.74(16)	C(13)-C(14)-C(15)-C(20)	-177.17(13)
C(2)-C(1)-C(10)-C(15)	-82.62(18)	C(19)-C(14)-C(15)-C(20)	2.3(2)
C(4)-C(1)-C(10)-C(15)	95.96(15)	C(11)-C(10)-C(15)-C(14)	-2.4(2)
C(15)-C(10)-C(11)-C(12)	0.9(2)	C(1)-C(10)-C(15)-C(14)	177.90(12)
C(1)-C(10)-C(11)-C(12)	-179.42(13)	C(11)-C(10)-C(15)-C(20)	177.13(13)
C(15)-C(10)-C(11)-C(16)	-178.45(13)	C(1)-C(10)-C(15)-C(20)	-2.6(2)

Symmetry transformations used to generate equivalent atoms: