m × 0.3 mm SE 52 glass capillary column, t_R 19.3 min (*Z*) and 19.7 min (*E*)): ^{1}H NMR (400 MHz, $CDCl_3$) 5.49 (ddq, J_d = 15 Hz, J_d = 7 Hz, J_q = 1 Hz, 1 H, H-3), 5.36 (dqd, J_d = 15 Hz, J_q = 7 Hz, J_d = 1 Hz, 1 H, H-4), 1.63 (d, J = 7 Hz, 3 H, CH_3), 1.22 (s, 12 H, CH_3), 1.04 (d, J = 7 Hz, 3 H, CH_3); ^{13}C NMR ($CDCl_3$) 133.2, 122.5, 82.9, 24.6, 18.0, 15.1. Anal. Calcd for $C_{11}H_{21}BO_2$: C, 67.36; H, 10.81. Found: C, 67.67; H, 10.92.

4,6-Dimethylnonan-5-ol (2a and 2c). To 2.0 g (10 mmol) of **3** in 10 mL of petroleum ether was added at 0 °C 1.0 g (10 mmol) of 2-methylpentanal.[10] The mixture was allowed to warm to room temperature and stirred for 14 h. After addition of 1.5 g (10 mmol) of triethanolamine in 5 mL of CH_2Cl_2 the resulting suspension was stirred for 2 h. The mixture was miltered through 45 g of silica gel and the product was eluted with CH_2Cl_2. Concentration afforded 2.1 g of a colorless oil which was taken up in 10 mL of methanol. This solution was stirred with 0.2 g of 5% palladium on charcoal for 1 day at 25 °C under 1 atm of hydrogen. The reaction was filtered, the filtrated concentrated, and the resulting oil distilled to yield 1.3 g (76%, 104–107 °C (14 torr)) of a 25:75 mixture of **2a** and **2b**.

For **2a**: ^{13}C NMR ($CDCl_3$) 81.0, 35.1, 33.0, 20.2, 16.5, 14.3. Anal. Calcd for $C_{11}H_{24}O$: C, 76.68; H, 14.04. Found: C, 76.34; H, 14.27.

For **2c**: ^{13}C NMR ($CDCl_3$) 79.0, 36.5, 35.7, 34.4, 34.4, 20.2, 20.0, 15.9, 14.3, 14.2, 12.7.

Acknowledgment. Support of this research by the National Institutes of Health (GM-31750) and the Robert A. Welch Foundation (F-626) is gratefully acknowledged.

Registry No. **1a**, 98757-88-1; **1b**, 82917-19-9; **1c**, 98757-89-2; **2a**, 98757-90-5; **2c**, 79237-71-1; **3**, 98704-47-3; 2-chloro-3-pentene, 1458-99-7; triisopropyl boranate, 5419-55-6; pinacol, 76-09-5; 2-methylpentanal, 123-15-9.

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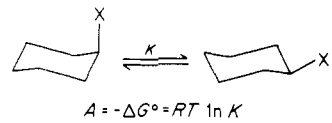
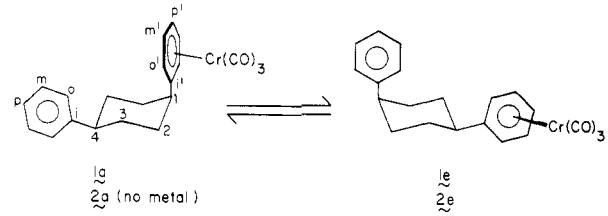
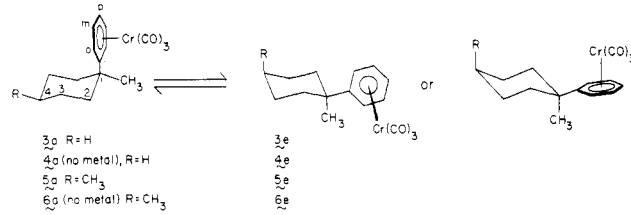
Scheme I



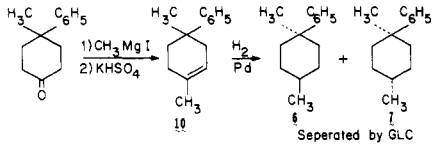
Scheme II



Scheme III



Scheme IV



The Conformational Energy of the (Benzene)chromium Tricarbonyl Substituent

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Among the many substituents whose equatorial preference[2] in cyclohexane ($\Delta G°$, Scheme I) has been recorded[3–5] there are a few metalloorganic groups such as $HgBr$,[3] $SnMe_3$,[6] $GeMe_3$,[6] and $PbMe_3$[6] but no complexes of transition elements. We report here the equatorial preference of the (benzene)chromium tricarbonyl [$C_6H_5Cr(CO)_3$] substituent, both by itself and when geminal to a methyl group, as determined by low-temperature ^{13}C NMR spectroscopy.

The equatorial preference of phenyl amounts to 2.87 kcal/mol.[7] Since this value is too large to be measured directly by low-temperature ^{13}C NMR spectroscopy (the favored methodology) and since we expected the corresponding $-\Delta G°$ value of the (benzene)chromium tricarbonyl substituent to be of the same order of magnitude, we decided to counterpoise the complexed phenyl group against an uncomplexed one by studying (*cis*-4-phenylcyclohexylbenzene)chromium tricarbonyl (**1**, Scheme II). The parent hydrocarbon **2**[8] was converted to **1** by treatment with chromium hexacarbonyl in refluxing dioxane, the major product (41.5%) being the monocomplex. The ^{13}C NMR chemical shifts of both the ligand **2** and the complex **1** at room temperature and at −100 °C are given in Table I, and the ratios of corresponding peaks are indicated in Table II. The assignments in the low-temperature spectrum of the ligand are based on known[7] spectra with equatorial and axial phenyl groups; knowledge of the ligand shifts then made possible assignment of most of the signals in the complex. The averaged equilibrium constant of 1.2 ± 0.1 corresponds to $-\Delta G° = 0.06 \pm 0.03$ kcal/mol at −100 °C; i.e., the equatorial preference of the complexed phenyl group is only marginally larger than that of the uncomplexed one.

This conclusion is reasonable in light of earlier calculations[9] which suggest that the equatorial phenyl ring lies in the symmetry plane of the cyclohexyl ring and the axial one is perpendicular to that plane (cf. Scheme II). In that case, the equatorial phenyl can be complexed sideways (apparently with little steric problem) and the axial one (likewise) from the outside face of the ring. (If there is any steric congestion, it must be about the same for the two different attitudes of the phenyl ring.)

(1) (a) University of North Carolina at Chapel Hill. (b) North Carolina State University.

(2) Alternatively called "conformational energy" or "A-value", cf. ref 3.

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0022-3263/85/1950-4978$01.50/0

Table I. Chemical Shifts (ppm in CD_2Cl_2 Except as Noted)

compd	C(1)	C(2)	C(3)	C(4)	Me-1	Me-4	ipso	ortho	meta	para
2[c]	40.2_2	29.9_4[a]	29.9_4[a]	40.2_2			146.2_2[b]	127.2_4	128.2_1	125.5_7
2[d]	35.1_5[e]	(30.0_8)[f]	(29.6_3)[f]	44.8_1[g]			144.1_9[e]	127.8_7[h]	128.4_8[h]	125.4_9[h]
							147.9_9[g]	127.8_7[h]	128.4_8[h]	125.4_9[h]
1[c]	(38.8_9)[i]	(29.7_3)[i]	(29.8_4)[i]	(39.8_3)[i]			117.3_2[j]	(91.5_3)[f,j]	91.9_9[f,j]	90.5_3[j]
							144.3_4[k]	(126.8_4)[f,k]	(128.1_9)[f,k]	125.6_6[k]
1a[d]	35.0_6[h]	29.7_6[h]	29.2_2	44.0_6[h]			147.5_3[k]	127.7_9[k]	128.5_4[k]	126.4_0[k]
							119.7_8[j]	93.3_8[j]	94.2_9[j]	93.0_6[j]
1e[d]	42.9_6	29.7_6[h]	30.2_3	35.0_6[h]			143.5_1[k]	127.0_9[k]	128.5_7[k]	125.6_4[k]
							119.4_6[j]	95.3_8[j]	95.3_9[j]	91.7_4[j]
3[c]	36.7_7	38.3_4	22.7_8	26.2_2	25.4_7		125.4_0[j]	91.2_1[j]	94.0_6[j]	94.9_4[j]
3e[d,l]	35.4_5	36.6_1	21.5_0	25.0_5	23.3_5		124.5_0[j]	91.0_1[j]	93.7_4[j]	94.9_3[j]
6[c]	38.2_1	37.6_4	31.5_2	32.9_0	35.0_5	22.1_8	147.7_1	126.4_3	128.3_0	125.1_4
7[c]	36.7_0	37.5_6	31.1_7	32.3_8	24.8_7	22.3_5	152.7_9	(125.2_0)[f]	(125.3_9)[f]	128.0_8
5[c]	30.7_7	36.6_3	30.4_8	32.8_5	36.6_9	20.6_8	123.0_3	89.8_8	96.0_1	95.9_2
5e[d]	*m*	36.2_1	27.4_1	26.5_0	25.1_2	17.2_4	*n*	92.0_8	94.1_7	95.5_1
5a[d]	*m*	38.3_8	31.2_2	32.5_7	37.6_1	22.6_7	121.0_1	89.4_3	98.6_0	97.8_5
10[o]	133.1_7	119.9_5	37.7_1	35.9_9	23.32	28.5_4	149.4_6	125.6_9	127.9_5	125.4_2
11[c]	44.0_7	34.5_6	34.5_6	44.0_7			147.3_9	126.8_2	128.3_4	125.9_5

[a] Misprinted in ref 8. [b] This value supersedes that given in ref 8. [c] At room temperature. [d] At −100 °C. [e] Carbon next to or of axial phenyl. [f] Uncertain assignments; may have to be inverted. [g] Carbon next to or of equatorial phenyl. [h] Signals of axial and equatorial phenyl groups not resolved. [i] The assignment of the 1/4 and the 2/3 carbons is uncertain. [j] Complexed phenyl. [k] Uncomplexed phenyl. [l] Only one peak at 31.9_0 could be detected for the minor isomer **3a** but was not assigned with certainty. [m] Signal could not be located. [n] Because of some artifacts in the 125–130 ppm region, the signal of the minor isomer could not be located with certainty. [o] Also C(5) 35.2_2 ppm and C(6) 28.0_9 ppm.

Table II. Area Ratios

	ipso	ortho	meta	para	C(2)	C(3)	C(4)	Me(1)	Me(4)	*K*
1e/1a	1.2_3[a]	1.1_8[a]	*b*	1.0_9[a]	*b*	1.2_6	1.2_4[c]			1.2 ± 0.1[d]
5a/5e	*e*	4.4_4	4.4_8	3.9_8	3.8_9	5.4_1	4.2_6	4.7_6	5.4_9	4.6 ± 0.6[d]

[a] Uncomplexed phenyl. [b] Not resolved. [c] Comparison of C(1) in **1e** with C(4) in **1a**. [d] Averaged area ratios. [e] Peak not used.

An entirely different conclusion is reached for the complex of 1-phenyl-1-methylcyclohexane[7] (**3**, Scheme III). It is known[7,9] that in this case, in the free ligand **4**, axial phenyl is preferred over equatorial by 0.32 kcal/mol because the addition of the geminal methyl group to the "perpendicular" axial phenyl causes no extra strain, whereas the insertion of axial methyl into the equatorial phenylcyclohexane causes a serious steric problem (cf. **4**, Scheme III). Complex **3**, in contrast to the free ligand **4**, appeared to be largely biased toward the equatorial phenyl side (chemical shifts at room temperature and of the major conformer at −100 °C are shown in Table I[10]). We therefore decided to counterpoise the system with a methyl group. The necessary ligand (**6**, Scheme III) was prepared as shown in Scheme IV[11] and complexed as before. The room and low-temperature (−100 °C) spectra of complex **5** are shown in Table I and the peak ratios are summarized in Table II, from which $1/K = 4.6 \pm 0.6$ and $\Delta G° = 0.52 \pm 0.05$ kcal/mol for the equation as written in Scheme III. Since $-\Delta G°_{Me} = 1.74 \pm 0.06$ kcal/mol,[12] the calculated $-\Delta G°$ for **3** is $1.74 - 0.52 = 1.22 \pm 0.08$ kcal/mol. Thus the introduction of the chromium tricarbonyl moiety into **4** to produce **3** causes a shift in equilibrium from the axial phenyl to the equatorial phenyl conformer, the change in free energy being about 1.5 kcal/mol. We ascribe this difference to the unfavorable free energy of complexation of the axial phenyl group from the side of the geminal methyl group (the alternative complexation from the side of the ring is probably even more severely sterically impeded); in contrast, the equatorial phenyl group can probably form a stable complex readily, with the chromium tricarbonyl moiety complexing either from the side (if the phenyl ring is in the symmetry plane of the cyclohexane ring) or from the top (if the phenyl ring is perpendicular to that plane), as shown in Scheme III.

In summary, these two results show that the effective "size" of a phenyl group may or may not be increased significantly by complexation with chromium tricarbonyl, depending on the molecular surroundings.

Experimental Section

Carbon-13 spectra were recorded on a Bruker Spectrospin WM-250 FT spectrometer at 62.896 MHz and proton spectra on the same instrument at 250 MHz or on a Varian XL-100 instrument at 100 MHz. All chemical shifts are recorded in ppm downfield from tetramethylsilane.

1,4-Dimethyl-*r*-1-phenylcyclohexan-*t*-4-ol (8) and 1,4-Dimethyl-*r*-1-phenylcyclohexan-*c*-4-ol (9). A solution of 12.6 g (0.066 mol) of 4-methyl-4-phenylcyclohexanone[13] in 20 mL of absolute ether was added to a solution of methylmagnesium iodide prepared from magnesium turnings (1.92 g, 0.08 mol) and methyl iodide (11.4 g, 0.08 mol) in 100 mL of absolute ether and refluxed for 1 h. It was then cooled, saturated NH_4Cl solution (50 mL) was carefully added, the ether layer was separated, and the aqueous layer was extracted with ether (3 × 20 mL). The combined ether solution was washed with saturated sodium chloride solution (50 mL), dried over anhydrous K_2CO_3, and concentrated to yield a white solid, which was shown by ^{13}C NMR to be a mixture of ca. 50% **8** and 50% **9**. The epimeric alcohol mixture was used in the next step without further purification: ^1H NMR ($CDCl_3$) (**8** and **9**) δ 1.11 (s, 3 H, Me), 1.18 (s, 3 H, Me), 1.23 (s, 3 H, Me), 1.30 (s, 3 H, Me), 1.35–1.72 (m, 2 × 6 H), 1.77–1.90 (m,

(10) The room-temperature and low-temperature ^{13}C NMR spectrum of **4** is reported in ref 7.

(11) The room-temperature ^{13}C NMR spectra of ligand **6** and its diastereomer **7** are recorded in Table I. The assignment of configuration is based on the downfield C(1) methyl shift and upfield ipso carbon phenyl shift in **6** relative to **7**. Moreover, the shifts of C(1) in **6** and **7** are very close to those of the correspondng conformers in **4** (38.7_0 and 36.7_6 ppm, respectively). Finally and most convincingly the shifts in **6** and **7**, respectively, are closely simulated by adding the shift parameters for equatorial Me-4 to the known[7] shifts for the phenyl axial and phenyl equatorial 1-methyl-1-phenylcyclohexane conformers.

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2 × 1 H), 2.00–2.15 (m, 2 × 2 H), 7.14–7.42 (m, 2 × 5 H); ^{13}C NMR ($CDCl_3$) 29.4_2, 30.1_9, 32.7_9, 33.1_8, 33.7_5, 35.7_2, 35.7_7, 36.8_8, 37.6_6, 69.4_1, 69.4_7, 125.3_7, 125.4_6, 125.9_7, 128.1_9, 128.2_8 ppm.

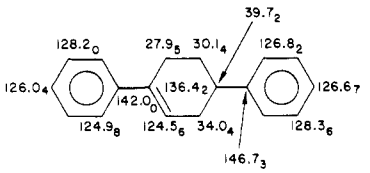
1,4-Dimethyl-4-phenylcyclohexene (10). The epimeric alcohol mixture (10 g) was mixed with 10 g of powdered $KHSO_4$ in a 100-mL round-bottom flask equipped with a Kugelrohr receiver bulb and placed in the Kugelrohr heater. The flask was heated at 150 °C for ca. 1 h which caused water droplets to collect in the receiver. The receiver was changed and aspirator vacuum applied to distill the olefin [bp 150–160 °C (20 mm)]. The product thus collected was redistilled to yield 8.67 g (95%) of olefin **10**: MS, *m/e* 186, 118, 117, 91; ^{1}H NMR ($CDCl_3$) δ 1.24 (s, 3 H), 1.58–1.60 (d, *J* = 3 Hz, 3 H), 1.68–1.81 (m, 2 H), 1.85–1.98 (m, 2 H), 2.02–2.15 (m, 1 H), 2.41–2.83 (m, 1 H), 5.39–5.45 (m, 1 H), 7.11–7.18 (br t, 1 H), 7.23–7.38 (m, 4 H); ^{13}C NMR ($CDCl_3$), see Table I.

1,*c*-4-Dimethyl-*r*-1-phenylcyclohexane (6) and 1,*t*-4-Dimethyl-*r*-1-phenylcyclohexane (7). The olefin **10** (6.5 g, 35 mmol) was dissolved in 50 mL of absolute ethanol in a hydrogenation bottle, 900 mg of 10% Pd on carbon was added, and the mixture was shaken under hydrogen (initial pressure 45 psi) in a Parr apparatus for 1 h. Filtering through a Celite pad, removing the solvent, and distillation of the residue [bp 140–145 °C (20 mm)] in a Kugelrohr yielded 6.1 g (92%) of **6** and **7** in a ratio of ca. 3:2 as shown by GC. This mixture was separated by preparative GLPC using an Apiezon-L column at 170 °C. Compound **6** emerged first followed by **7**.

cis-Isomer **6**: MS, *m/e* 188, 173, 131, 118, 105, 91, and 32. Anal. Calcd for $C_{14}H_{20}$: M^+, 188.157. Found: M^+, 188.157. ^{1}H NMR ($CDCl_3$) δ 0.76–0.82 (d, *J* = 7 Hz, 3 H), 0.96–1.04 (m, 1 H), 1.14 (s, 3 H), 1.2–1.62 (m, 6 H), 2.22–2.50 (m, 2 H),7.1–7.4 (m, 5 H); ^{13}C NMR, see Table I.

trans-Isomer **7**: MS, *m/e* 188, 173, 131, 118, 105, 91. Anal. Calcd for $C_{14}H_{20}$: M^+, 188.157. Found: M^+, 188.157. ^{1}H NMR ($CDCl_3$) δ 0.96–1.00 (d, *J* = 7 Hz, 3 H), 1.1–1.9 (m, 9 H), 1.28 (s, 3 H), 7.2–7.44 (m, 5 H); ^{13}C NMR, see Table I.

Alternate Synthesis of *cis*- (2) and *trans*-1,4-Diphenylcyclohexane (11). The epimeric alcohol mixture of *t*-1,*c*-4-diphenyl-*r*-1-cyclohexanol and *t*-1,*t*-4-diphenyl-*r*-1-cyclohexanol[8] was heated with $KHSO_4$ as described above for **10** to yield 1,4-diphenylcyclohex-1-ene (**12**) in ca. 90% yield, mp 101–103 °C (lit.[14] mp 102 °C) after vacuum distillation in a Kugelrohr: ^{1}H NMR δ 1.96–2.22 (m, 2 H), 2.3–2.7 (m, 4 H), 2.8–3.0 (m, 1 H), 6.18–6.30 (m, 1 H, olefinic), 7.2–7.5 (m, 10 H, Ph); ^{13}C NMR ($CDCl_3$) (in ppm)



12

Catalytic reduction of **12** in absolute ethanol at 45 psi initial hydrogen pressure with 10% by weight of 10% Pd–C yielded *cis*-1,4-diphenylcyclohexane (**2**) of ca. 95% purity as an oil. However, hydrogenation in 95% ethanol using the same catalyst at atmospheric pressure yielded a mixture of **2** and **11**.[15] From the cis–trans mixture the trans (**11**) compound crystallizes from ethanol, mp 171–172 °C (lit.[16] mp 170–172 °C; ^{13}C NMR of **2** and **11**; see Table I.

***cis*-(1,4-Diphenylcyclohexane)chromium Tricarbonyl (1).** *cis*-1,4-Diphenylcyclohexane (**2**) (0.294 g, 1.24 mmol) and $Cr(CO)_6$ (0.824 g, 4.12 mmol) in 20 mL of dioxane were heated at reflux in an argon atmosphere for 20 h. Removal of solvent at reduced pressure left a yellow-green solid, which was dissolved in anhydrous ether and filtered through a filter-aid pad to remove the excess chromium hexacarbonyl. Evaporation of ether at reduced pressure left a residue (0.833 g), which was loaded onto a column of silica gel HF-254 packed in 20% ethyl acetate in hexane. Elution with the same solvent gave complexation product **1** (0.193 g, 42% yield) as a yellow-green oil: IR (CCl_4) 1980, 1910, 1550, 650, 630 cm^{-1}; ^{1}H NMR (CCl_4) δ 1.80 (m, 8 H), 2.55 (br m, 1 H), 2.87 (br m, 1 H), 5.11 (s, 5 H), 7.14 (s, 5 H). ^{13}C NMR, see Table I.[17]

Chromium tricarbonyl complexes **3** and **5** were prepared similarly and were characterized by ^{13}C NMR spectroscopy (Table I).[17]

Acknowledgment. Part of this work was supported by NSF Grant CHE-802088.

Registry No. **1**, 98859-22-4; **2**, 21072-41-3; **3**, 98859-23-5; **4**, 828-45-5; **5**, 98859-24-6; **6**, 98859-17-7; **7**, 98859-18-8; **8**, 98859-19-9; **9**, 98859-20-2; **10**, 98859-21-3; **11**, 21072-42-4; **12**, 10470-07-2; 4-methyl-4-phenylcyclohexanone, 18932-33-7; *t*-1,*c*-4-diphenyl-*r*-1-cyclohexanol, 93783-02-9; *t*-1,*t*-4-diphenyl-*r*-1-cyclohexanol, 93783-03-0.

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(17) Since these arenechromium tricarbonyl compounds are air-sensitive amorphous substances of low volatility which are not readily obtained in a state of analytical purity, they were not submitted for elemental analysis.

A Facile Procedure for Oxidative Cleavage of Enolic Olefins to the Carbonyl Compounds with Ruthenium Tetraoxide (RuO_4)

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Oxidative cleavage of a carbon–carbon double bond to the corresponding carbonyl compounds is an essential operation in organic synthesis.[1] Ozonolysis is accepted as the most general method for this purpose, but this technique involves the tedious feature that the reaction is carried out at low temperature by bubbling an excess amount of ozone.[2] On the other hand, among the high valent metal salts suitable for the oxidative cleavage of olefins,[3] ruthenium tetraoxide (RuO_4) is promising with respect to its high efficiency[4] and is already employed in the conversion of simple olefins to the carbonyl compounds.[5] However, to date no systematic study of this application to an enolic system has appeared except for one case attempted in the cleavage of C=C bond of steroidal enamines.[6] In this paper, we disclose a versatile procedure for the oxidative cleavage of enolic olefins including enol ethers, enol acetates, and enamines to give

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