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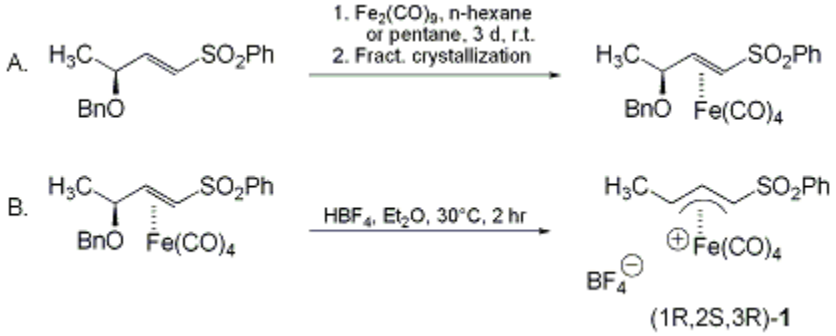
September 2014: The paragraphs above replace the section "Handling and Disposal of Hazardous Chemicals" in the originally published version of this article. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

DOI:10.15227/orgsyn.078.0189

Organic Syntheses, Coll. Vol. 10, p.672 (2004); Vol. 78, p.189 (2002).

(+)-(1R,2S,3R)-TETRACARBONYL[(1-3η)-1-(PHENYLSULFONYL)-BUT-2-EN-1-YL]IRON(1+) TETRAFLUOROBORATE

[Iron(1+), tetracarbonyl[(1,2,3-η)-1-(phenylsulfonyl)-2-butenyl]-, stereoisomer, tetrafluoroborate(1)-]



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Checked by Matthew J. Schnaderbeck and William R. Roush.

1. Procedure

A. (+)-(E,1R,3S)-Tetracarbonyl[(3-(benzyloxy)-1-(phenylsulfonyl)-η²-but-1-ene]iron(0). A flame-dried, 250-mL Schlenk-flask equipped with a septum, magnetic stirring bar and a balloon filled with carbon monoxide (Note 1) is charged with 3.03 g (10.0 mmol) of (−)-(E,S)-3-(benzyloxy)-1-butenyl phenyl sulfone (Note 2) and 4.73 g (13.0 mmol) of nonacarbonyldiiron [$Fe_2(CO)_9$] (Note 3). The reagents are suspended in 150 mL of anhydrous hexane (Note 4) and the orange suspension is stirred for 3 days at room temperature under an atmosphere of carbon monoxide and with exclusion of light (Note 5). The reaction mixture is diluted with anhydrous diethyl ether (ca. 100 mL) (Note 6) and transferred via canula to an air-free filter containing a short column of Celite (Note 7) by means of a flame-dried inert gas frit (Note 8) and filtered under an atmosphere of argon. The bright yellow filtrate is collected in a 1000-mL, flame-dried Schlenk-flask (Note 5). The filter column is washed with anhydrous diethyl ether (Note 6) until the filtrate becomes colorless. The clear yellow solution of the neutral complex is concentrated under reduced pressure in a room temperature water bath to between a third and a quarter of the original volume by means of a medium pressure vacuum pump (Note 9) and is then fractionally crystallized at −25°C in a freezer. The pale yellow precipitate is washed with anhydrous hexane pre-cooled to −25°C (Note 4) under an atmosphere of argon and dried under reduced pressure at room temperature by means of a high vacuum pump (Note 9) with the exclusion of light (Note 5) to afford 1.88-3.05 g (40-65%) of a moderately air sensitive pale yellow crystalline solid (Notes 10, 11).

B. (+)-(1R,2S,3R)-Tetracarbonyl[(1-3η)-1-(phenylsulfonyl)but-2-en-1-yl]iron(1+) tetrafluoroborate **1**. A flame-dried, 50-mL Schlenk-flask equipped with a septum and an argon balloon is charged with 1.88 g (4.0 mmol) of (+)-(E,1R,3S)-tetracarbonyl[(1-2η)-3-(benzyloxy)-1-(phenylsulfonyl)-but-1-ene] iron(0) and the complex is dissolved in 15 mL of anhydrous diethyl ether (Note 6) under an atmosphere of argon. The bright yellow solution is taken up into a syringe under an atmosphere of argon and filtered through a PTFE-syringe filter into a flame-dried, 100-mL Schlenk flask equipped with a magnetic stirring bar, a septum and an argon balloon (Note 12). The solution is diluted with anhydrous diethyl ether (Note 6) to give a total volume of 50-70 mL and then warmed to 30°C by means of a water bath. Fluoroboric acid, (HBF_4), 0.7 mL (4.8 mmol) of a 54% solution of HBF_4 in diethyl ether (Note 13) is added dropwise by means of a syringe to the rapidly stirring solution. After ca. 2 hr, the precipitate is filtered off under an atmosphere of argon using an inert gas frit. The precipitate is washed with anhydrous diethyl ether (Note 6) until the filtrate becomes colorless (3 × 10-20 mL). The residue is dried for ca. 12 hr under reduced pressure at room temperature using a high vacuum pump (Notes 8, 9) to yield 1.73 g (96%) of a pale yellow solid (Note 14). Normally the complex is obtained in

spectroscopically and analytically pure form (by ^{1}H and ^{13}C NMR, elemental analysis) and can be used without further purification for nucleophilic addition reactions. If necessary, the complex can be further purified by re-precipitation from nitromethane solution with the addition of excess cold diethyl ether . The dry complex should be stored at −25°C in a freezer under an atmosphere of argon and may be handled for short periods in air.

2. Notes

1. Carbon monoxide was purchased from Linde, Germany (99%) and was used without further purification.
2. Prepared according to the accompanying procedure: Enders, D.; von Berg, S.; Jandeleit, B. *Org. Synth.* **2002**, *78*, 177.
3. According to ref. 3, nonacarbonyldiiron [$Fe_2(CO)_9$] was synthesized by photochemical dimerization of pentacarbonyliron [$Fe(CO)_5$] in a mixture of glacial acetic acid and acetic acid anhydride (10:1) at room temperature employing a Dema irradiation apparatus with a Philips HPK 125 W or TQ 150 W medium pressure mercury lamp. The material should be stored at −25°C in a freezer and handled under an atmosphere of argon. Pentacarbonyliron [$Fe(CO)_5$] was a gift from BASF AG, Germany and was used without further purification.
4. Hexane was purified by distillation from calcium hydride under argon.
5. Aluminum foil should be wrapped around the Schlenk flask to exclude sunlight.
6. diethyl ether was purified by distillation from sodium and benzophenone.
7. Celite was purchased from Fluka, Germany and dried in an oven for 4 hr at ca. 110°C. The dried material was degassed three times in an inert gas frit prior to use by an evacuation/argon purge cycle. After this procedure the Celite was compressed to a ca. 4-cm deep layer and then covered by an 1-cm layer of previously dried sea sand (Riedel de Haën, Netherlands) to avoid a disturbance to the Celite layer during manipulations.
8. Aluminum foil should be wrapped around the inert gas frit to exclude sunlight.
9. An additional effective cooling trap should be installed to condense any of the highly toxic pentacarbonyliron [$Fe(CO)_5$].
10. Initially, a diastereomeric mixture of olefinic iron complexes (de ≈ 70%) is obtained from which the desired major diastereomer can be separated in a highly diastereo- and enantiomerically enriched form following the crystallization procedure described. A second crop can be obtained from the mother liquor to increase the chemical yield. However, additional fractions may not be as diastereomerically pure as the first fraction, giving rise to cationic complexes of lower enantiomeric purity in the next step.
11. The compound shows the following analytical and spectroscopic data: R_f = 0.43 (0.25-mm silica gel on glass, diethyl ether/light petroleum, 1:2); mp: 103°C (dec.); de = ee > 99% (by ^{1}H NMR, 500 MHz, signals: $CHCH_3$, ortho-C-H); $[\alpha]_D^{26}$ +171.8 (benzene, *c* 1.05); ^{1}H NMR (500 MHz, C_6D_6) δ: 0.91 (d, 3 H, J = 6.1), 3.04 (qdd, 1 H, J = 6.1, 5.8, 0.3), 3.29 (dd, 1 H, J = 10.4, 5.8), 3.79 (d, 1 H, J = 12.1), 3.86 (dd, 1 H, J = 10.2, 0.3), 3.93 (d, 1 H, J = 12.1), 6.87-7.13 (m, 8 H), 7.85-7.91 (m, 2 H) ; ^{13}C NMR (125 MHz, C_6D_6) δ: 21.73, 57.96, 66.70, 70.15, 76.31, 127.84, 128.04, 128.23, 128.47, 129.13, 132.54, 137.97, 142.66, 207.25 ppm ; IR (KBr) cm^{-}: 3085, 3056, 3032 (w, Ar-C-H), 2969, 2873 (w, CH, CH_2, CH_3), 2103 (vs, apical-Fe-CO), 2045, 2022, 1988 (vs, Fe-CO), 1585, 1496, 1479 (vw, Ar-C=C), 1446 (m), 1385, 1377 (w, CH_3), 1326 (m), 1300 (s, S=O), 1262 (w-m), 1191 (w), 1144 (s, S=O), 1084 (s, C-O-C), 1041, 1026 (m), 807, 752, 734, 718, 689 (m), 624 (vs), 591, 562 (s) ; IR (hexane) cm^{-}: 2104, 2035, 2026, 2002 (vs, Fe-CO). MS (70 eV): m/z (%): 414 (3) [M^{+}.-2CO], 386 (4) [M^{+}.-3CO], 359 (20), 358 (99) [M^{+}.-4CO], 303 (3) [M^{+}.+1-$Fe(CO)_4$], 268 (14), 267 (100) [358-C_7H_7], 250 (14), 239 (17), 217 (4) [358-$SO_2C_6H_5$], 198 (10), 186 (10), 184 (12), 161 (27), 143 (3) [$H_2SO_2C_6H_5^{+}$], 141 (2) [$SO_2C_6H_5^{+}$], 134 (12), 133 (53), 107 (6) [$C_7H_8O^{+}$], 91 (79) [$C_7H_7^{+}$],77 (20) [$C_6H_5^{+}$], 65 (16) [$C_5H_5^{+}$], 56 (63) [Fe^{+}], 55 (10), 53 (17), 51 (10) ; calculated for $C_{21}H_{18}FeO_7S$ (M_r: 470.28): C 53.63, H 3.86, found C 53.60, H 3.89.
12. A stainless steel filtration device and PTFE-filters (Satorius, Germany, diameter: 25 mm, pore size: 0.45 µm) were used to purify the solution by removing paramagnetic impurities from the neutral complex. If the solution is not filtered, paramagnetic impurities may be found in the solidified complex but these should not affect reactivity of the resulting cationic complex.
13. The 54% solution of HBF_4 in diethyl ether was purchased from Merck, Germany and was used without further purification. The acid solution should be stored in a refrigerator under an atmosphere of argon to avoid colorization and any loss of quality.

14. The compound shows the following analytical and spectroscopic data: mp: 163°C (crystals yellowed), 173°C (dec.); de > 99% (3-syn/3-anti: >> 99: << 1 by ^{1}H NMR, 500 MHz, signals: $CHCH_3$, $CH\text{-}CHSO_2$, $CHCH_3$); ee > 99%; $[\alpha]_D^{21}$ +169.1 (acetone, *c* 1.14); ^{1}H NMR (500 MHz, CD_3NO_2) δ: 2.13 (d, 3 H, J = 6.4), 4.64 (dd, 1 H, J = 10.1, 0.6), 4.93 (dqd, 1 H, J = 12.4, 6.4, 0.6), 6.23 (dd, 1 H, J = 12.4, 10.1), 7.72-7.80 (m, 2 H), 7.82-7.91 (m, 1 H), 8.07-8.14 (m, 2 H) ; better H NMR data were obtained in d_6-acetone: [1] ^{1}H NMR (500 MHz, d_6-acetone) δ: 2.20 (d, 3 H, J = 6.1), 5.10 (dd, 1 H, J = 10.0, 0.6), 5.17 (dq, 1 H, J = 12.4, 6.3), 6.59 (ddd, 1 H, J = 12.4, 10.0, 0.6), 7.72-7.82 (m, 2 H), 7.82-7.9 (m, 1 H), 8.1-8.2 (m, 2 H) ; ^{13}C NMR (125 MHz, CD_3NO_2) δ: 20.77, 73.73, 90.58, 97.65, 129.46, 131.54, 136.61, 139.57, 195.35, 196.10, 197.43, 197.67 ; IR (KBr) cm^{-}: 3067, 3007 (w, Ar-CH), 2929, 2857 (w), 2162, 2142, 2125, 2100, 2030, 2006 (s-vs, Fe-CO), 1642, 1585, 1521 (w, Ar-C=C), 1448 (m), 1386 (w, CH_3), 1303, 1148 (s, S=O), 1084, 1057 (vs, br.), 810, 756, 727, 719, 689 (w), 628, 612, 594 (s), 555 (w); IR (CH_2Cl_2) cm^{-}: 2102 (vs, Fe-CO), 2032, 2000 (vs, Fe-CO) .MS (70 eV): m/z (%): 446 (2) [M^{+}.+1], 321 (2), 306 (3) [321-CH_3], 278 (13), 250 (13) [278-CO], 196 (25), 195 (14) [M^{+}.-BF_4^{-}, -$Fe(CO)_4$], 186 (7), 184 (21), 161 (7), 143 (4) [$H_2SO_2C_6H_5^{+}$], 141 (3) [$SO_2C_6H_5^{+}$], 133 (9), 129 (13), 126 (15), 125 (28), 115 (6), 108 (6), 107 (24), 105 (6), 97 (10), 95 (9), 94 (9), 93 (11), 91 (19), 79 (18), 78 (20), 77 (41) [$C_6H_5^{+}$], 56 (49) [Fe^{+}], 55 (100), 53 (18), 50 (8), 48 (8), 43 (10), 41 (15), 39 (24) ; calculated for $C_{14}H_{11}BF_4FeO_6S$ (M_r: 449.95): C 37.37, H 3.14, found C 36.93, H 2.76.

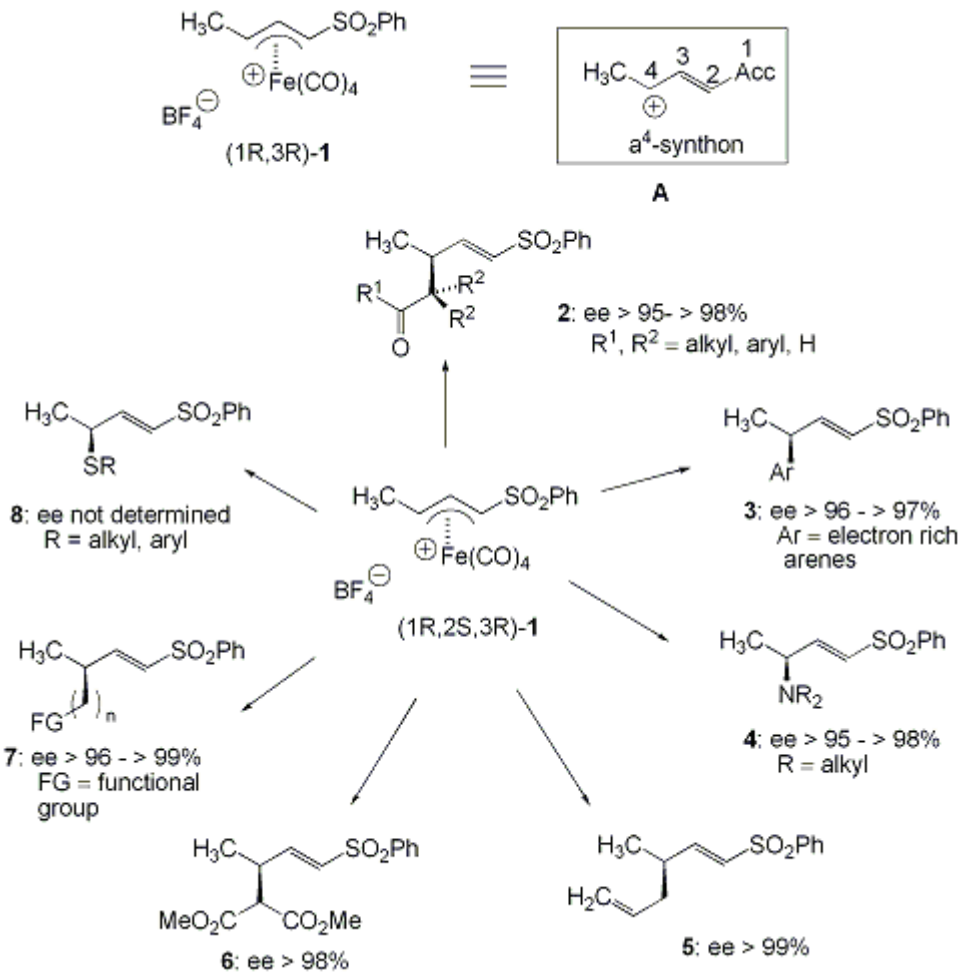
Waste Disposal Information

All toxic materials were disposed of in accordance with "Prudent Practices in the Laboratory"; National Academy Press; Washington, DC, 1995.

3. Discussion

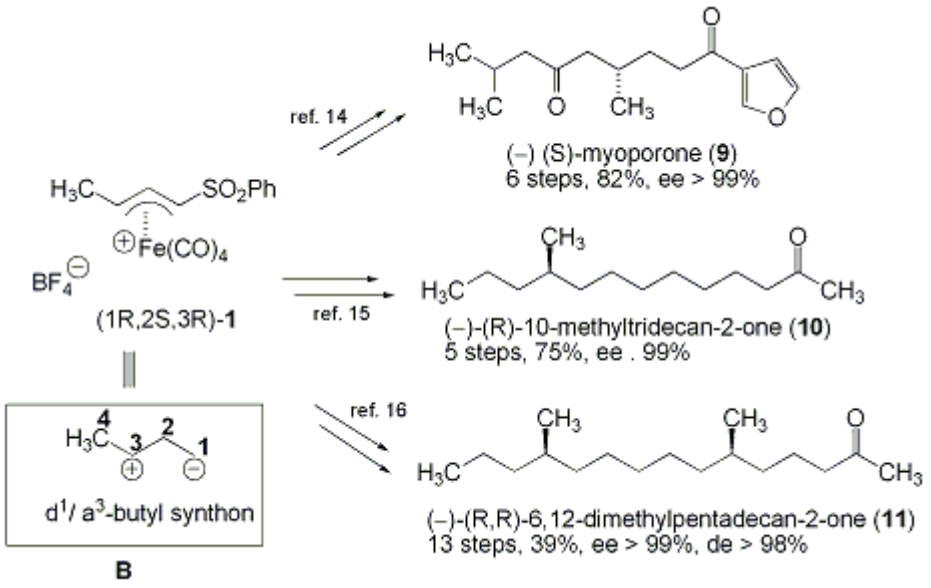
Among the various carbon-carbon and carbon-hetero atom bond forming reactions promoted or catalyzed by transition metals, allylic substitution via electrophilic π-allyl-complexes is of utmost importance. Studies focused on the synthetic potential of alkyl or aryl substituted (η^3-allyl)$Fe(CO)_4$(1+) complexes have shown that nucleophilic attack by soft carbon and hetero atom nucleophiles preferentially proceeds regioselectively at the less or syn-substituted allyl terminus.[4] Additionally, polar effects on the regioselectivity of this reaction caused by electron-withdrawing functionalities (e.g., CO_2R, $CONR_2$) have been examined by the submitters' group,[4] Green, et al.,[5,6] and Speckamp, et al.[7] It has been demonstrated that the reaction affords allyl-coupled addition products with complete γ-regioselectivity with respect to the electron-withdrawing functionality. During the submitters' efforts[3] devoted to developing a useful methodology for the synthesis of highly enantiomerically enriched compounds via iron-mediated allylic substitutions, the "chirality transfer" approach turned out to be a practical solution. Based on the chiral pool precursor lactic acid, an efficient approach to the phenylsulfonyl-functionalized planar chiral (η^3-allyl)$Fe(CO)_4$(1+) complex (1R,2S,3R)-**1**in virtually diastereo- and enantiomerically pure form (de, ee > 99%) has been developed.[8] Complex (1R,2S,3R)-**1**represents a synthetic equivalent of an a^4-synthon **A**with planar chirality (Scheme 2)allowing homologous (1,5)-Michael additions[9] or an "Umpolung" of classical d^4-chemistry.[10] Regioselective addition of carbon and heteroatom nucleophiles to (1R,2S,3R)-**1** (Scheme 2) provides an efficient access to highly enantiomerically enriched alkenyl sulfones with a wide range of substitution patterns at the allylic position; a class of compounds which is of increasing importance.[11] As depicted in Scheme 2, (1R,2S,3R)-**1**can be combined with various nucleophiles.[8,12,13] These reactions proceed with virtually no loss of chirality information from central (C-O) to planar (C-Fe) and back to central chirality (C-C or C-X) affording products **2**-**8** with high enantiomeric purity (ee > 95 - > 99%) and overall retention (double inversion) with respect to the starting material. In addition, the reaction proceeds with complete γ-regioselectivity and conservation of the (E)-double bond geometry leading to highly functionalized molecules of well-defined stereochemistry.

Scheme 2



Furthermore, the planar complex (1R,2S,3R)-**1**also represents the synthetic equivalent of a d^1/a^3-butyl synthon **B**(Scheme 3). This stems from its electrophilic reactivity in the γ-position and its nucleophilic reactivity, after reductive hydrogenation and metalation, α- to the sulfonyl group, with subsequent removal of the latter. The potential bifunctionality allows a flexible sequential functionalization of the butyl-backbone of **1**. Because of their origin from enantiopure building blocks bearing methyl substituents (e.g., isoprenoids, alanine, lactic acid derivatives) many naturally occuring compounds possess methyl-branched carbon atom skeletons. Scheme 3 demonstrates some achievements made in natural product synthesis making use of complex **1**. Key steps in the syntheses of these natural products have been the nucleophilic addition of silyl enol ether or allyl-silane to **1**, respectively.[14,15,16] Starting from complex **1**, all methyl-branched natural products synthesized (**9**, **10**, **11**) have been obtained from readily accessible materials with their naturally occurring absolute configuration in excellent overall yields and in virtually enantio- and/or diastereomerically pure form (**9**, **10**: ee > 99%, **11**: ee > 99%, de > 98%).[17]

Scheme 3



References and Notes

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Appendix
Chemical Abstracts Nomenclature (Collective Index Number); (Registry Number)

(+)-(1R,2S,3R)-Tetracarbonyl[(1-3η)-1-(phenylsulfonyl)-but-2-en-1-yl]iron(1+) tetrafluoroborate:
Iron(1+), tetracarbonyl[(1,2,3-η)-1-(phenylsulfonyl)-2-butenyl]-, stereoisomer, tetrafluoroborate(1−) (13); (162762-06-3)

(+)-(E,1R,3S)-Tetracarbonyl[(3-benzyloxy)-1-(phenylsulfonyl)-η^2-but-1-ene]iron (0):
Iron, tetracarbonyl[[[[(2-3η)-1-methyl-3-(phenylsulfonyl)-2-propenyl]oxy]methyl]benzene]-, stereoisomer (13); (168431-28-5)

(−)-(E,S)-3-(Benzyloxy)-1-butenyl phenyl sulfone:
Benzene[[[1-methyl-3-(phenylsulfonyl)-2-propenyl]oxy]methyl]-, [S-(E)]- (13); (168431-27-4)

Carbon monoxide (8,9); (630-08-0)

Nonacarbonyldiiron:
Iron, tri-μ-carbonylhexacarbonyldi- (Fe-Fe) (8,9); (15321-51-4)

Fluoroboric acid:
Borate(1−), tetrafluoro-, hydrogen (8,9); (16872-11-0)