synchrotron or inverse Compton emission from the jet already observed in the radio band (23). Unfortunately, current knowledge of the jet at radio wavelengths does not allow discriminating between the two processes.

To have such a clear polarimetric signal, the magnetic field has to be coherent over a large fraction of the emission site (5). Such a coherent magnetic-field structure may indicate a jet origin for the gamma rays above 400 keV (24). In addition, because the gamma rays emitted in BH x-ray binaries are generally thought to be emitted close to the BH horizon (7, 25), and because the synchrotron photons we observed in the hard tail are too energetic to be effectively self-Comptonized, these observations might be evidence that the jet structure is formed in the BH vicinity, possibly in the Compton corona itself. Another possibility is that the gamma rays are produced in the initial acceleration region in the jet, as observed at higher energies by the Fermi Large Area Telescope from the microquasar Cygnus X-3 (26).

The spectrum above 400 keV is consistent with a power law of photon index 1.6 ± 0.2. This means that this spectrum, if due to synchrotron or inverse Compton emission, is caused by electrons whose energy distribution is also a power law with an index of 2.2 ± 0.4 (27), consistent with the canonical value for shock-accelerated particles p = 2. Synchrotron radiation at MeV energies implies also that the electron energy, for a magnetic field of 10 mG, which is reasonable for this kind of system (28), would be around a few TeV (27, 29). Inverse Compton scattering of photons off these high-energy TeV electrons, whose lifetime due to synchrotron energy loss is about 1 month (27), could also be the origin of the TeV photons detected from Cygnus X-1 with the Major Atmospheric Gamma-ray Imaging Cerenkov telescope experiment (30) and possibly also the gamma rays claimed by Astrorivelatore Gamma ad Immagini Leggero/Light Imager for Gamma-Ray Astrophysics (31).

The position angle (PA) of the electric vector, which gives the direction of the electric field lines projected onto the sky, is 140° ± 15°. This is at least 100° away from the compact radio jet, which is observed at a PA of 21° to 24° (32). Such deviations between the electric field vector and jet direction are also found in other jet sources, such as Active Galactic Nuclei (33) or the galactic source SS433 (34).

References and Notes

25. In brief, a Compton polarimeter measures the polarization dependency of the differential cross section for Compton scattering, where linearly polarized photons scatter preferentially perpendicularly to the incident polarization vector. By examining the scatter-angle azimuthal distribution of the detected photons, a sinuosidal signal is obtained from which the PA and the PI with respect to a 100% polarized source can be derived. Gamma-ray polarization measurements are particularly difficult, the main difficulty being the exclusion of systematic/detector effects in the azimuthal Compton events distribution. To exclude these systematic effects, we followed the process detailed in (19). We only considered events that interacted once in the upper CsI crystal layer, integral Soft Gamma-Ray Imaging (sensitive in the 15–1000-keV band), and once in the lower CsI layer, Pixelated CsI telescope (sensitive in the 200–1000-keV band), and whose reconstructed energy was in the 250–7000-keV energy range. These events were automatically selected on board through a time coincidence algorithm. The maximal allowed time window was set to 3.8 µs during our observations, which span between 2003 and 2009, for a total exposure of more than 5 million seconds, which is ~58 days.

Selective, Nickel-Catalyzed Hydrogenolysis of Aryl Ethers

Alexey G. Sergeev and John F. Hartwig*

Selective hydrogenolysis of the aromatic carbon-oxide (C=O) bonds in aryl ethers is an unsolved synthetic problem important for the generation of fuels and chemical feedstocks from biomass and for the liquefaction of coal. Currently, the hydrogenolysis of aromatic C=O bonds requires heterogeneous catalysts that operate at high temperature and pressure and lead to a mixture of products from competing hydrogenolysis of aliphatic C-O bonds and hydrogenolysis of the arene. Here, we report hydrogenolyses of aromatic C-O bonds in alky1 aryl and diaryl ethers that form exclusively amines and alcohols. This process is catalyzed by a soluble nickel carbene complex under just 1 bar of hydrogen at temperatures of 80 to 120°C; the relative reactivity of ether substrates scale as Ar-OAr >> Ar-OCHMe (Ar, Aryl; Me, Methyl). Hydrogenolysis of lignin model compounds highlights the potential of this approach for the conversion of refractory aryl ether biopolymers to hydrocarbons.

S

elective hydrogenolysis of aromatic carbon-oxide (C=O) bonds (scission by reaction with hydrogen to form CH and OH bonds in their place) is challenging because of the strength and stability of these linkages (I); yet, this process is important for the conversion of oxygen-rich lignocellulosic plant biomass to deoxygenated fuels and commercial chemicals (2–5). Whereas the exclusively aliphatic C-O bonds in cellulose can be cleaved with hydrolysis and dehydrogenation (J), the aromatic C-O bonds in lignin cannot undergo these processes and have resisted selective cleavage by hydrogen (2, 4). In addition, brown coal’s polymeric network contains aromatic C-O bonds inherited from lignocellulosic biomass, and the liquefaction of these bonds could facilitate the liquefaction of this carbon source and its conversion to arene feedstocks (6). A general, mild method for reductive clea-
age of unactivated aromatic C-O bonds would
furthermore expand the utility of alkoxy and ar-
yloxy substituents as removable directing groups
(7) that can influence synthetic transformations
of aromatic systems.

In contrast to hydrogcnolysis of benzyl ethers,
which proceeds selectively under mild conditions
over heterogeneous catalysts (8), the cleavage of
C-O bonds in aryl ethers requires high tempera-
tures and pressures (over 250°C and 30 bar)
and occurs with poor selectivities (1, 2); typical
product mixtures include saturated cyclic hydro-
carbons and cycloalkanols from hydrogenation of
the arene rings and concurrent aliphatic C-O bond
cleavage (1, 2, 9–12). This arene reduction wastes
designed and renders the reaction less useful for
producing aromatic feedstocks (2, 4). Alternative
methods for the cleavage of aryl ethers that avoid
this side reaction require stoichiometric alkali
metals (13) or electrocatalytic hydrogcnolysis (14),
both of which are expensive and difficult to con-
duct on a large scale. Here, we report the selec-
tive, reductive cleavage of various aromatic C-O bonds with hydrogen catalyzed by a single solu-
bile nickel catalyst under mild conditions (15).
Hydrogcnolysis with this catalyst proceeds with-
out concurrent arene ring hydrogenation and re-
duction of aliphatic C-O bonds.

To realize a selective hydrogcnolysis of ar-
omc C-O bonds, we envisioned a reaction that
involves insertion of a discrete transition metal
complex into the aromatic C-O bond and reaction
of the resulting intermediate with hydrogen
to yield arenes and alcohol. We anticipated that
the low reactivity of homogeneous catalysts
toward hydrogenation of aromatic rings (16)
would prevent competitive formation of cyclo-
alkanes and cycloalkanols from such a process.
Nickel complexes known to activate aromatic
C-O bonds in the presence of aliphatic C-O bonds
(17–21) were the starting point for our catalyst
development.

Catalytic reactions involving aromatic C-O bond
cleavage with nickel complexes were first
reported by Wenkert for cross-coupling of aryl
ethers with Grignard reagents to form biaryl com-
ounds (17, 18). This reactivity has been de-
veloped further in recent years with less aggressive
carbon nucleophiles and improved catalysts (19–21).
However, the extension of such reactivity to the
hydrogcnolysis of C-O bonds is challenging be-
cause hydrogen is less reactive than main group
carbon nucleophiles, and the hydrogcnolysis or
hydrogenation of typically unreactive moieties
can lead to reduction of the catalyst itself to form

**Table 1.** Hydrogcnolysis of diaryl ethers. The reactions were conducted in closed glass reaction vessels
filled with hydrogen at 1 bar gauge pressure at 24°C. After the time listed, no further changes in conversion
were observed. The yields of arenes and phenols formed by the bond cleavage shown were measured with
gas chromatography after acidification and aqueous work up.

<table>
<thead>
<tr>
<th>Entry</th>
<th>Diaryl ether</th>
<th>Ni, mol%</th>
<th>T, °C</th>
<th>Time, h</th>
<th>Conversion, %</th>
<th>Arene, %</th>
<th>Phenol, %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td></td>
<td>20</td>
<td>120</td>
<td>16</td>
<td>100</td>
<td>99</td>
<td>99</td>
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<tr>
<td>2</td>
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<td>10</td>
<td>120</td>
<td>32</td>
<td>87</td>
<td>82</td>
<td>87</td>
</tr>
<tr>
<td>3</td>
<td></td>
<td>5</td>
<td>120</td>
<td>32</td>
<td>59</td>
<td>59</td>
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<td>20</td>
<td>120</td>
<td>32</td>
<td>85</td>
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</tbody>
</table>

*Anisole (65%) and benzene (23%). †3-Methoxyphenol (83%) and phenol (3%). ‡Trifluoromethylbenzene (64%)
and toluene (23%); benzene (4%) as a side product. §Trifluoromethylbenzene (68%) and toluene (19%).
‖Anisole (4%) as a side product. ¶Phenol (17%) as a side product.
range of aryl and benzyl ethers with main group hydride donors, we explored the use of hydrogen—a cheaper, cleaner, and milder reagent—for the reduction of aryl ethers (tables S3 and S4). Indeed, the combination of Ni(COD)$_2$ and SIPr·HCl (20 mol %) as catalyst and NaO$tBu$ (2.5 equiv.) as base effected the selective hydrogenolysis of various diaryl ethers with only 1 bar pressure of H$_2$ as base, which was observed in any of the experiments. As noted earlier, this complete selectivity for reduction of the C-O bonds over the aryl π-bonds contrasts with that for hydrogenolysis of aryl ethers over heterogeneous catalysts, which typically leads to a mixture of arenes, cycloalkanes, phenols, and cycloalkanols (1, 2, 9–12).

The nickel catalyst is also active under comparable conditions for the selective hydrogenolysis of aryl alkyl ethers to form arenes and aliphatic alcohols (Table 2). For example, the hydrogenolysis of 1-methoxynaphthalene, 2-alkoxynaphthalenes, and 4-alkoxybiphenyls under hydrogenolysis occurred faster with unsubstituted diphenyl ether and diaryl ethers bearing an electron-rich and electron-poor arenes. Hydrogenolysis of alkyl aryl ethers.

### Table 2. Hydrogenolysis of alkyl aryl ethers.

<table>
<thead>
<tr>
<th>Entry</th>
<th>Alkyl Aryl Ether</th>
<th>T, °C</th>
<th>Time, h</th>
<th>Additive (1 equiv.)</th>
<th>Conversion, %</th>
<th>Arene, %</th>
<th>AlkOH, %</th>
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</thead>
<tbody>
<tr>
<td>1</td>
<td>OAlk</td>
<td>120</td>
<td>16</td>
<td>-</td>
<td>100</td>
<td>95</td>
<td>98</td>
</tr>
<tr>
<td>2</td>
<td>Alk = nHexyl</td>
<td>120</td>
<td>16</td>
<td>-</td>
<td>89</td>
<td>99</td>
<td>nq*</td>
</tr>
<tr>
<td>3</td>
<td>Alk = Methyl</td>
<td>80</td>
<td>16</td>
<td>AlMe$_3$</td>
<td>100</td>
<td>98</td>
<td>nq</td>
</tr>
<tr>
<td>4</td>
<td>OMe</td>
<td>120</td>
<td>16</td>
<td>-</td>
<td>72</td>
<td>72</td>
<td>nq</td>
</tr>
<tr>
<td>5</td>
<td></td>
<td>100</td>
<td>16</td>
<td>AlMe$_3$</td>
<td>100</td>
<td>99</td>
<td>nq</td>
</tr>
<tr>
<td>6</td>
<td>OAlk</td>
<td>120</td>
<td>32</td>
<td>-</td>
<td>85</td>
<td>85</td>
<td>85</td>
</tr>
<tr>
<td>7</td>
<td>Alk = Methyl</td>
<td>120</td>
<td>32</td>
<td>AlMe$_3$</td>
<td>60</td>
<td>59</td>
<td>nq</td>
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<tr>
<td>8</td>
<td>Ph</td>
<td>100</td>
<td>32</td>
<td>AlMe$_3$</td>
<td>65</td>
<td>65</td>
<td>nq</td>
</tr>
</tbody>
</table>

*nq, not quantified.

### Table 3. Hydrogenolysis of benzyl ethers.

<table>
<thead>
<tr>
<th>Entry</th>
<th>Alkyl Benzyl Ether</th>
<th>Ni, mol%</th>
<th>Additive (1 equiv.)</th>
<th>Conversion, %</th>
<th>Arene, %</th>
<th>R$^2$OH, %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Bu</td>
<td>10</td>
<td>-</td>
<td>100</td>
<td>99</td>
<td>93</td>
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<tr>
<td>2</td>
<td>Bu</td>
<td>20</td>
<td>-</td>
<td>1</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>3</td>
<td>Bu</td>
<td>20</td>
<td>AlMe$_3$</td>
<td>100</td>
<td>99</td>
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<tr>
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<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>5</td>
<td>Me</td>
<td>20</td>
<td>AlMe$_3$</td>
<td>100</td>
<td>96</td>
<td>nq</td>
</tr>
</tbody>
</table>

*nq, not quantified. †32 hours.
lectivity, we conducted the hydrogenolysis of diphenyl ether and 2-methoxynaphthalene in the presence of a 300-fold excess of mercury with respect to the catalyst. No decrease in conversion or product yields was observed (23). Although such data are suggestive, rather than fully conclusive, of the homogeneity versus heterogeneity of the catalyst, the absence of poisoning by mercury is consistent with our proposal that the distinct selectivities result from the action of a homogeneous catalyst (22).

Having identified a catalyst for the reductive cleavage of simple diaryl ethers, we tested the ability of this system to catalyze the hydrogenolysis of an oligomeric aryl ether. Hydrogenolysis of the oligomeric phenylene oxide containing eight aromatic C-O bonds (Fig. 3A) in the presence of 10 mol % of the catalyst per C-O bond under 1 bar of hydrogen at 120°C in m-xylene resulted in complete depolymerization to give benzene, phenol, and resorcinol in good total yield.

Lastly, we studied the hydrogenolysis of various aromatic and benzylic C-O bonds that constitute roughly 75% of all the intermonomer linkages in lignin, one of the most stable biopolymers in nature (Fig. 3, B to D) (2, 5). Cleavage of each of these ether linkages with a single catalyst would illustrate the potential to conduct the catalytic depolymerization and valorization of lignin to form aromatic products (4). The diaryl ether models of one of the most recalcitrant lignin linkages, the 4-O-5 linkage, were cleaved under 1 bar of hydrogen at 120°C in m-xylene to yield anisole, benzene, and phenols in moderate yields in the presence of 20 mol % of the catalyst generated from Ni(COD)₂ and SIPr·HCl (Fig. 3B). Hydrogenolysis of the α-O-4 lignin model compound proceeded with just 5 mol % of the Ni-SIPr catalyst at 80°C under 1 bar of hydrogen to afford 3,4-dimethoxytoluene and 2-methoxyphenol in nearly quantitative yields (Fig. 3C). In agreement with previous results, cleavage of the β-O-4 model under the basic conditions of our system proceeded without catalyst to give guaiacol in 89% yield (Fig. 3D) (30).

Available data do not allow one to distinguish between several possible mechanisms for the nickel-catalyzed hydrogenolysis of aryl ethers, but several mechanisms can be envisioned. The nickel center that cleaves the aromatic C-O bond could be a nickel hydride, a neutral nickel(0) complex, or an anionic nickelate species. The favorable effect of the strong base in the hydrogenolysis could lead to the formation of anionic nickel complexes that are more active for the cleavage of the C-O bonds or for activation of coordinated dihydrogen. Although these mechanistic issues are yet to be resolved, and turnover numbers of the catalyst must be improved, the results from this work have demonstrated that the selective cleavage of aromatic C-O bonds in the presence of other C-O bonds can be conducted without reduction of the arene units by using a widely available metal and the cheap, mild, and atom-economical reductant hydrogen.

Fig. 2. (A) Relative reactivity of aryl and benzyl ethers toward hydrogenolysis of C-O bonds catalyzed by Ni(COD)₂ and SIPr. Selective hydrogenolysis of (B) diphenyl ether in the presence of 4-tert-butylbenzyl methyl ether; (C) 2-methoxynaphthalene in the presence of 4-tert-butylbenzyl methyl ether; and (D) diphenyl ether in the presence of 4-tert-butylanisole.

Fig. 3. Hydrogenolysis of (A) bis(m-phenoxypheynyl)benzene. (B) A model of the 4-O-5 linkage in lignin. (C) A model of the α-O-4 linkage in lignin. (D) A model of the β-O-4 linkage in lignin.

References and Notes
7. We define a directing group as a substituent, usually containing a basic heteroatom, attached to the aromatic ring that enables functionalization of the arene by...
reaction of an organometallic reagent at a specific position, usually ortho to the directing group, followed by the reaction of the metallated aromatic compound with an electrophile. For application of these groups in directed ortho-lithiation see (31).


15. Stoichiometric hydrogenolysis of aromatic C-O bond in a palladium pincer complex was reported in (12).


23. Materials and methods are available as supporting material on Science Online.

24. We observed formation of cyclohexane and cyclohexene in the reaction of Ni(COD)2 and PCy2 with H2 in the absence of diphenyl ether under the same conditions (23).


27. Ni(COD)2 and PCy2 were reported to catalyze silane-induced cleavage of polyalicyclic aromatic ethers (methoxynaphthalenes) and alkyl aryl ethers containing directing groups ortho to the C-O bond (33, 34).

28. No reaction was observed in the absence of Ni(COD)2, as shown through control experiments on the cleavage of 4-tet-butylenyl methyl ether with hydrogen in the presence of AlMe3 (1 equiv.), SiPr2HCl (0.4 equiv.), and NaO(Bu)2 (2.5 equiv.) in m-xylene at 120°C for 32 hours (23).

29. For example, heterogeneous hydrogenolysis of alkyl benzyl ethers proceeds selectively in the presence of diaryl ethers over Pd(OH)2/C (25).


36. We thank the BP for financial support of this project through the Energy Biosciences Program and T. Rauchfuss for helpful discussions. A provisional patent has been filed on the methods presented herein.

Supporting Online Material
www.sciencemag.org/cgi/content/full/332/6028/439/DC1
Materials and Methods
SOM Text
Figs. S1 to S3
Tables S1 to S6
References
15 November 2010; accepted 11 March 2011
10.1126/science.1200437

High-Performance Electrocatalysts for Oxygen Reduction Derived from Poly-aniline, Iron, and Cobalt

Gang Wu,1 Karen L. More,2 Christina M. Johnston,1 Piotr Zelenay1*

The prohibitive cost of platinum for catalyzing the cathodic oxygen reduction reaction (ORR) has hampered the widespread use of polymer electrolyte fuel cells. We describe a family of non–precious metal catalysts that approach the performance of platinum-based systems at a cost sustainable for high-power fuel cell applications, possibly including automotive power. The approach uses poly-aniline as a precursor to a carbon-nitrogen template for high-temperature synthesis of catalysts incorporating iron and cobalt. The most active materials in the group catalyze the ORR at potentials within ~60 millivolts of that delivered by state-of-the-art carbon-supported platinum, combining their high activity with remarkable performance stability for non–precious metal catalysts (700 hours at a fuel cell voltage of 0.4 volts) as well as excellent four-electron selectivity (hydrogen peroxide yield <1.0%).

Thanks to the high energy yield and low environmental impact of hydrogen oxidation, the polymer electrolyte fuel cell (PEFC) represents one of the most promising energy conversion technologies available today. Of the many possible applications, ranging from sub-watt remote sensors to residential power generators in excess of 100 kW, automotive transportation is especially attractive. PEFCs promise major improvements over gasoline combustion, including better overall fuel efficiency and reduction in emissions (including CO2). The spectacular progress in fuel cell technology notwithstanding, a large-scale market introduction of fuel cell–powered vehicles continues to face various challenges, such as the lack of hydrogen infrastructure and the technical issues associated with PEFC performance and durability under the operating conditions of an automotive power plant. The high cost of producing PEFCs represents the most formidable challenge and has driven much of the applied and fundamental fuel cell research in recent years.

According to the latest cost analysis, the fuel cell—more precisely, the fuel cell stack—is responsible for more than 50% of the PEFC power system cost (1, 2). Although a state-of-the-art PEFC stack uses several high-priced components, the catalysts are by far the most expensive constituent, accounting for more than half of the stack cost. Because catalysts at both the fuel cell anode and cathode are based on platinum (Pt) or platinum alloys, their cost is directly linked to the price of Pt in the volatile and highly monetized precious metal market. The precious metal catalyst is the only fuel cell stack component that will not benefit from economies of scale, and an increase in the demand for fuel cell power systems is bound to drive up the already high price of Pt, about $1830 per troy ounce at present ($2280 per troy ounce at its maximum in March 2008) (3). Thus, PEFCs are in need of efficient, durable, and inexpensive alternatives to Pt and Pt-based catalysts.

Ideally, Pt should be replaced at both fuel cell electrodes; however, its substitution at the cathode with a non–precious metal catalyst would have comparatively greater impact, because the slow oxygen reduction reaction (ORR) at this electrode requires much more Pt than the faster hydrogen oxidation at the anode. As a consequence, the development of non–precious metal catalysts with high ORR activity has recently become a major focus of PEFC research (4–8).

The Pt replacement candidates that have attracted the most attention have been synthesized by heating precursors comprising nitrogen, carbon, and graphically abundant transition metals, iron and cobalt (M = Co and/or Fe) in particular (9–14). Although the nature of the active ORR catalytic sites in such N-M-C catalysts continues to be at the center of an ongoing debate (6, 7, 10, 15), there is no doubt that the ORR performance of N-M-C catalysts strongly depends on the type of nitrogen and transition-metal precursors used, heat treatment temperature, carbon support morphology, and synthesis conditions.

We recently initiated a research effort to develop non–precious metal catalysts that combine high ORR activity with good performance stability, originally concentrating on materials obtained without heat treatment. The polypyrrole (Ppy)-Co-C system prepared this way showed respectable performance durability for a non–precious metal...
ERRATUM

Post date 11 June 2011

Reports: “Selective, nickel-catalyzed hydrogenolysis of aryl ethers” by A. G. Sergeev and J. F. Hartwig (22 April, p. 439). There were two errors introduced during proofs. The third sentence of the first paragraph should read, “In addition, brown coal’s polymeric network contains aromatic C-O bonds inherited from lignocellulosic biomass, and the hydrogenolysis of these bonds could facilitate the liquefaction of this carbon source and its conversion to arene feedstocks (6).” The last sentence of the last paragraph should read, “Although these mechanistic issues have yet to be resolved, and turnover numbers of the catalyst must be improved, the results from this work have demonstrated that the selective cleavage of aromatic C-O bonds in the presence of aliphatic C-O bonds can be conducted without reduction of the arene units by using a widely available metal and the cheap, mild, and atom-economical reductant hydrogen.”
Selective, Nickel-Catalyzed Hydrogenolysis of Aryl Ethers
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