

Exhibit A

**IN THE UNITED STATES DISTRICT COURT
FOR THE EASTERN DISTRICT OF VIRGINIA
Alexandria Division**

AMERICAN CHEMICAL SOCIETY,

Plaintiff,

v.

SCI-HUB d/b/a WWW.SCI-HUB.CC,
JOHN DOEs 1-99,

Defendants.

No. 1:17-cv-00726-LMB-JFA

DECLARATION OF DAVID E. WESLOW

I, David E. Weslow, declare and state as follows:

1. I am a partner with the law firm of Wiley Rein LLP, and I am currently an active member in good standing of the Bars of Maryland and the District of Columbia. I am over the age of eighteen and competent to testify to the matters set forth herein. Unless otherwise indicated, I have personal knowledge of the facts stated herein and am prepared, if necessary, to testify to the statements herein at any hearing or trial relating to this matter or the issues or facts discussed herein.

2. Our firm is counsel of record for Plaintiff American Chemical Society (“ACS”) in this matter, and I make this Declaration in support of Plaintiff’s Motion for Default Judgment.

3. I have reviewed the online records maintained by the United States Copyright Office and identified more than 9,000 copyrights owned by ACS.

4. The copyrights owned by ACS include works contained in 45 publications identified in Exhibit E to Plaintiff’s First Amended Verified Complaint.

5. The Pirated/Spoofed Website operated by Defendant Sci-Hub includes a nearly identical copy of pages appearing on the legitimate website operated by ACS, acs.org, including display of at least 32 publications published by ACS and maintained behind a paywall on acs.org. True and accurate screenshots of the Pirated/Spoofed Website page containing access to the ACS copyrighted material for each of these publications are attached hereto as Attachment 1.

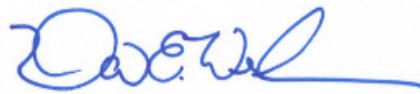
6. Each of the 32 publications includes multiple ACS copyrighted works as follows:

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7. In total, the Pirated/Spoofed Website operated by Defendant Sci-Hub infringes on at least 2,225 registered copyrights owned by ACS.

I declare under the penalty of perjury as required by 28 U.S.C. Section 1746 that to the best of my knowledge, the foregoing is true and correct. Executed in Washington, D.C., on September 1, 2017.



David E. Weslow

ATTACHMENT 1



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Article

One-Step Conversion from Core-Shell Metal-Organic Framework Materials to Cobalt and Nitrogen Co-Doped Carbon Nano-Polyhedra with Hierarchically Porous Structure for Highly Efficient Oxygen Reduction

Zhaowen Hu, Zhengping Zhang, Zhilin Li, Meiling Dou, and Feng Wang

ACS Appl. Mater. Interfaces, Just Accepted Manuscript • Publication Date (Web): 28 Apr 2017

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High-Throughput Investigation of a Lead-Free AlN-Based Piezoelectric Material, $(\text{Mg,Hf})\text{Al}_{1-x}\text{N}$ Hung H. Nguyen,^{*,†} Hiroyuki Oguchi,[‡] Le Van Minh,[†] and Hiroki Kuwano[‡][†]Department of Robotics, Tohoku University, 980-8577 Sendai, Japan[‡]Advanced Institute for Materials Research (AIMR), Tohoku University, 980-8577 Sendai, Japan

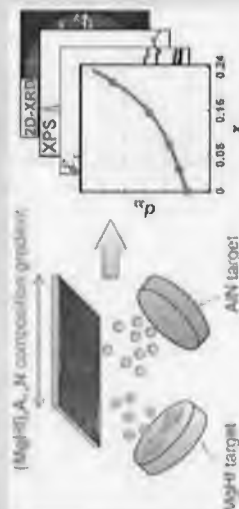
Supporting Information

ABSTRACT: We conducted a high-throughput investigation of the fundamental properties of $(\text{Mg,Hf})\text{Al}_{1-x}\text{N}$ thin films ($0 < x < 0.24$) aiming for developing high-performance AlN-based piezoelectric materials. For the high-throughput investigation, we prepared composition-gradient $(\text{Mg,Hf})\text{Al}_{1-x}\text{N}$ films grown on a Si(100) substrate at 600 °C by cosputtering AlN and Mg/Hf targets. To measure the properties of the various compositions at different positions within a single sample, we used characterization techniques with spatial resolution. X-ray diffraction (XRD) with a beam spot diameter of 1.0 mm verified that Mg and Hf had substituted into the Al sites and caused an elongation of the c-axis of AlN from 5.00 Å for $x = 0$ to 5.11 Å for $x = 0.24$. In addition, the uniaxial crystal orientation and high crystallinity required for piezoelectric materials to be used as application devices were confirmed. The piezoelectric response microscope indicated that this c-axis elongation increased the piezoelectric coefficient almost linearly from 1.48 pm/V for $x = 0$ to 5.19 pm/V for $x = 0.24$. The dielectric constants of $(\text{Mg,Hf})\text{Al}_{1-x}\text{N}$ were investigated using parallel plate capacitor structures with $\sim 0.07 \text{ mm}^2$ electrodes and showed a slight increase by substitution. These results verified that $(\text{Mg,Hf})\text{Al}_{1-x}\text{N}$ is a promising material for piezoelectric-based application devices, especially for vibrational energy harvesters.

KEYWORDS: Mg/Hf codoped AlN, piezoelectric material, combinatorial approach, piezoelectric coefficient, dielectric constant

INTRODUCTION

In the development of lead-free piezoelectric materials that can be used as alternative to lead zirconate titanate (PZT), AlN has drawn attention due to high electromechanical

Table 1. AlN-Based Piezoelectric Materials ($\text{M}_1\text{Al}_{1-x}\text{N}$)

M	max x	d_{31} at max x, pm/V	d_{31} increase rate, pm/V per atomic %	type of addition
(pure AlN)		3.9		

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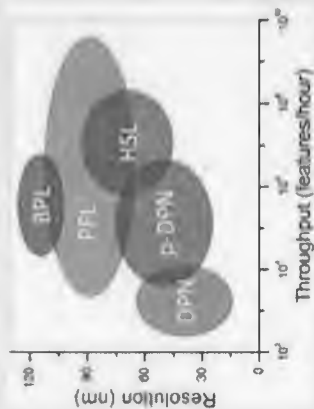
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Recent Advances in Cantilever-Free Scanning Probe Lithography: High-Throughput, Space-Confinement Synthesis of Nanostructures and Beyond

Qiyuan He, Chaoliang Tan, and Hua Zhang*

Center for Programmable Materials, School of Materials Science and Engineering, Nanyang Technological University, 50 Nanyang Avenue, Singapore 639798

ABSTRACT: Scalability is the major challenge for scanning probe lithography (SPL). Recently developed cantilever-free scanning probe technologies provide a solution to the issue of scalability by incorporating massive arrays of polymer pens, which fundamentally overcome the low-throughput nature of SPL. The further development of cantilever-free SPL brings up a variety of applications in electronics, biology, and chemical synthesis. In this Perspective, we highlight the space-confined synthesis of complex nanostructures enabled by different types of cantilever-free SPL technologies.



The development of nanoscience and nanotechnology demands the combination of versatile nanomaterials and capable nanofabrication techniques.¹ In particular, direct-write lithography offers advantages including the fabrication of arbitrary features in the nanometer scale, which is particularly useful in proof-of-concept and prototyping nanotechnologies.² The precise control of scanning probe microscopy (SPM) at the nanometer scale promises a wide

cantilever-free SPL and its applications in the space-confined synthesis of nanostructures.

In this Perspective, we discuss recent progress on high-throughput cantilever-free SPL and its applications in the space-confined synthesis of nanostructures.

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Affinity binding of EMR2 expressing cells by surface-grafted chondroitin sulfate B

Anouck L. S. Burzava, Marek Jasieniak, Michaela P. Cockshell, Claudine S. Bonder, Frances J. Harding, Hans Joerg Griesser, and Nicolas H. Voelcker

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How DNA-encoded libraries are revolutionizing drug discovery

With the bar-coding technology, drugmakers leverage the chemistry of large numbers
By *Bethany Halford*
(pp. 28-33)

FEATURES



Science & Technology Putting distillation out of business in the chemical industry

Purifying chemicals without heat would go a long way toward reducing global energy consumption and pollution (pp. 18-21)



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Nucleotide Excision Repair Lesion-Recognition Protein Rad4 Captures a Pre-Flipped Partner Base in a Benzo[a]pyrene-Derived DNA Lesion: How Structure Impacts the Binding Pathway

Hong Mu,[†] Nicholas E. Geacintov,[‡] Jung-Hyun Min,[‡] Yingkai Zhang,^{‡,§} and Suse Broyde^{*,†}

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Supporting Information

ABSTRACT: The xeroderma pigmentosum C protein complex (XPC) recognizes a variety of environmentally induced DNA lesions and is the key in initiating their repair by the nucleotide excision repair (NER) pathway. When bound to a lesion, XPC flips two nucleotide pairs that include the lesion out of the DNA duplex, yielding a productively bound complex that can lead to successful lesion excision. Interestingly, the efficiencies of NER vary greatly among different lesions, influencing their toxicity and mutagenicity in cells. Though differences in XPC binding may influence NER efficiency, it is not understood whether XPC utilizes different mechanisms to achieve productive binding with different lesions. Here, we investigated the well-repaired 10R-(+)-*cis*-anti-benzo[a]pyrene-N²-dG (*cis*-B[a]P-dG) DNA adduct in a duplex containing normal partner C opposite the lesion. This adduct is derived from the environmental pro-carcinogen benzo[a]pyrene and is likely to be encountered by NER in the cell. We have extensively investigated its binding to the yeast XPC orthologue, Rad4, using umbrella sampling with restrained molecular dynamics simulations and free energy calculations. The NMR solution structure of this lesion in duplex DNA has shown that the dC complementary to the adducted dG is flipped out of the DNA duplex in the absence of XPC. However, it is not known whether the “pre-flipped” base would play a role in its recognition by XPC. Our results show that Rad4 first captures the displaced dC, which is followed by a tightly coupled lesion-extruding pathway for productive binding. This binding path differs significantly from the one deduced for the small *cis*-syn cyclobutane pyrimidine dimer lesion opposite mismatched thymines [Mu, H., et al. (2015) *Biochemistry*, 54(34), 5263–7]. The possibility of multiple paths that lead to productive binding to XPC is consistent with the versatile lesion recognition by XPC that is required for successful NER.



CHEMICAL REVIEWS

Editorial

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Introduction: Super-Resolution and Single-Molecule Imaging

The invention of the light microscope propelled science forward by granting researchers the ability to magnify objects and study features with resolution well below that of the naked eye. However, despite centuries of improvements to imaging systems, optical microscopes could never surpass the fundamental diffraction limit of light, which prevents features smaller than roughly half a wavelength (several hundreds of nanometers) from being resolved.¹ Previously, this resolution limit was circumvented with near-field optics: light was squeezed through a nanosized aperture or focused to the end of a sharpened tip.^{2,3} Yet, this resolution enhancement came at the price of low signals, difficult aperture or tip fabrication, the need for a scanning probe geometry, and the perturbative nature of the tip upon the sample.⁴

However, in the last three decades, optical microscopy has been transformed, first through the first observation of single molecules,⁵ and then through the rapid expansion of a suite of far-field imaging techniques known collectively as super-resolution microscopy.^{6–10} Unlike near-field imaging, the current class of super-resolution techniques is based on far-field optics; through elegant optical tricks, each achieves resolution well below the wavelength of light. The rapid expansion and acceptance of super-resolution microscopy as a transformative technology was marked in 2014 when the Nobel Prize in Chemistry was awarded to Eric Betzig, Stefan Hell, and W. E. Moerner, three pioneers of single-molecule and super-resolution imaging. Thus, this thematic issue of *Chemical Reviews* dedicated to super-resolution imaging is timely, and we intend it to provide perspective on how the field has developed over a relatively short time, as well as look to the future for new developments and emerging applications.

This thematic issue has two primary objectives: first, to introduce the main classes of super-resolution imaging, including instrumentation, sample preparation, and data analysis; and second, to highlight the broad range of applications that have benefitted from these emerging

the models and data analysis strategies that have emerged to interpret the large data sets acquired during localization-based super-resolution imaging and obtain accurate representations of the underlying sample.¹² Finally, Landes and co-workers describe both theory and applications in which single-molecule localization is extended with single-particle tracking to follow single-molecule dynamics in real time, with applications in both biophysics and materials science.¹³

In contrast to single-molecule localization microscopy, another approach to achieve super-resolution optical imaging uses patterned illumination to effectively squeeze the diffraction limit to subwavelength dimensions. Widengren and co-workers present one such strategy, stimulated emission depletion (STED) microscopy, in a review that details the instrumentation, sample preparation, and applications.¹⁴ STED is a two-beam technique that uses nonlinear optics to shape the excitation beam into an effective subdiffraction-limited excitation volume, leading to subdiffraction-limited resolution in a confocal geometry.⁹ An alternative beam patterning approach is structured illumination microscopy (SIM). Here, a patterned excitation source is introduced to the sample, transforming high-frequency spatial information from the sample into low-frequency content that can be collected through the imaging optics of the system and then mathematically recovered during image processing.^{15,16} Incorporating nonlinearities like saturation into SIM significantly increases the resolution in super-resolution structured illumination microscopy (SR-SIM).¹⁷ While each of the super-resolution optical imaging techniques offer significant advantages on their own, the review from Xu and co-workers explores how careful sample preparation and experimental design allow the various forms of super-resolution imaging to be correlated with other imaging modalities, such as electron microscopy or atomic force microscopy, to obtain further nanoscopic information.¹⁸

The remaining reviews in this special issue are dedicated to the applications that benefit from achieving optical resolution



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public stronger."

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Managing Chemical Wastes in the High School Lab<https://www.acs.org/content/aact/en/periodical.html>

Rheology and Phase Behavior of Carbon Dioxide and Crude Oil Mixtures

Ruifen Hu, John P. Crawshaw,* J. P. Martin Trusler, and Edo S. Boek

Qatar Carbonate and Carbon Storage Research Centre (QCCSRC), Department of Chemical Engineering, Imperial College London, South Kensington Campus, London SW7 2AZ, United Kingdom

ABSTRACT: The rheology of Zuata heavy crude oil, saturated with carbon dioxide, was studied at a temperature of 50 °C and pressures up to 220 bar. Observations of phase behavior were also reported and used to interpret the rheological data. The crude oil is very viscous and non-Newtonian at ambient pressure, but when brought into equilibrium with CO₂, the non-Newtonian behavior was weakened and eventually disappeared at high CO₂ pressures. When diluted with 10 and 30 wt % toluene, the diluted crude oils and their mixtures with CO₂ behaved as Newtonian fluids. The CO₂-saturated mixture of the crude oil samples showed an exponential decrease in viscosity with increasing CO₂ pressure but an increase in viscosity at higher pressures. During observation through a view cell, the CO₂ dissolution caused a swelling effect on the original crude oil. When saturated with CO₂, the swelling effect also occurred on the 10 wt % diluted crude oil but the volume of the oil-rich phase was decreased at higher pressures. However, for the 30 wt % diluted crude oil, a second liquid phase was observed on top of the oil-rich phase, at pressures higher than the CO₂ critical point. The mixture viscosity was inversely proportional to the CO₂ solubility.

1. INTRODUCTION

Carbon dioxide (CO₂) released by the massive consumption of fossil fuels contributes significantly to global warming and extreme weather. As one of the final products of burning hydrocarbon fuels, its high stability and considerable concentration in the Earth's atmosphere makes CO₂ one of most important greenhouse gases. The surplus of CO₂ is caused by our extensive use of fossil fuels, which emits huge amounts of CO₂ into the atmosphere, and deforestation, which reduces the number of trees that absorb CO₂ during photosynthesis.¹ Immediate actions are required to control CO₂ emission, to prevent us from losing our security and prosperity by extensive climate change.

To reduce the CO₂ emission, a range of solutions based on different principles has been proposed, including carbon capture and storage (CCS). CCS is a family of technologies and

for four different CO₂-saturated heavy crude oils were measured and correlated at different temperatures and pressures.⁴ Similar studies can also be found in refs 5–11. In addition, the viscosity of CO₂ and alkane-diluted crude oil was reported by Li et al.¹²

Numerous papers report the phase behavior of CO₂ and crude oil mixtures. Similar to the mixture of CO₂ with alkanes, CO₂ and crude oil mixtures show liquid–liquid or liquid–liquid–vapor equilibria at temperatures and pressures lower than the CO₂–crude oil minimum miscibility point. The phase boundaries are determined by the crude oil composition, because CO₂ extracts the light and intermediate components in the crude oil to generate a second liquid phase on top of the oil-rich phase.^{13,14}

Moreover, as reported by many papers,^{6–9,15} CO₂ solubility in crude oil normally increases with increasing pressure but decreases with increasing temperature. The pressure sensitivity of CO₂ solubility at high pressures depends upon the phase of

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Optically Multiresponsive Heteroleptic Platinum Dithiolene Complex with Proton-Switchable Properties

Salahuddin Attar,[†] Davide Espa,[†] Flavia Artizzu,[†] Luca Pilia,[†] Angela Serpe,[†] Maddalena Pizzotti,[§] Gabriele Di Carlo,[§] Luciano Marchiò,^{||} and Paola Deplano^{*,†}

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Supporting Information

ABSTRACT: Both linear- and nonlinear-optical properties of $\text{Bu}_4\text{N}[\text{Pt}(\text{L}1)(\text{L}2)]$ ($\text{L}1$, $\text{L}1 = [4',5',6][1,4]$ -dithiino[2,3-*b*]quinoxaline-1',3'-dithiolato; $\text{L}2 = (R)$ - α -MBA dithiooxamidate, where (R) - α -MBA = (R) -(+)- α -methylbenzyl) upon HCl addition at room temperature change dramatically: the color turns from deep blue to green; the luminescence switches from deep red to green; the nonlinear-optical response (first hyperpolarizability) increases by a factor of 12. Thus, **1** behaves as a unique multiresponsive optical switch whose properties can be followed by the naked eye.

Changes of the linear-optical (L.O; absorption and emission of light)¹ as well nonlinear-optical (NLO)² properties in response to external stimuli are of current scientific interest and for several applications in devices.^{3–5} Molecules suitable for second-order NLO mostly consist of an electron-donor moiety connected to an electron-acceptor moiety by a conjugated π

refinements (Tables S1 and S2 and Figures S4–S6) are described.

The molecular structure of **1** comprises the complex anion $[\text{Pt}(\text{L}1)(\text{L}2)]^-$ and the Bu_4N^+ cation (Figure 1).

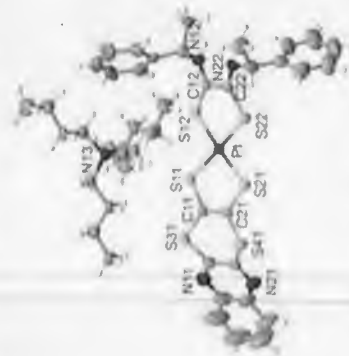


Figure 1. Molecular structure of **1** with thermal ellipsoids depicted at the

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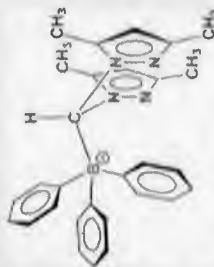
Monoanionic, Bis(pyrazolyl)methylborate $[(\text{Ph}_3\text{B})\text{CH}(3,5\text{-(CH}_3)_2\text{Pz}_2)]^-$ as a Supporting Ligand for Copper(II)-ethylene, *cis*-2-Butene, and Carbonyl Complexes

Shawn G. Ridlen, Naveen V. Kulkarni, and H. V. Rasika Dias*

Department of Chemistry and Biochemistry, The University of Texas at Arlington, Arlington, Texas 76019, United States

Supporting Information

ABSTRACT: The monoanionic bidentate ligand $[(\text{Ph}_3\text{B})\text{CH}(3,5\text{-(CH}_3)_2\text{Pz}_2)]^-$ has been prepared from lithium bis(pyrazolyl)methane and triphenylborane. This useful new ligand is closely related to the well-established bis(pyrazolyl)borate and bis(pyrazolyl)methane ligands but has key differences to both analogues as well. The ethylene, *cis*-2-butene, and carbon monoxide adducts $[(\text{Ph}_3\text{B})\text{CH}(3,5\text{-(CH}_3)_2\text{Pz}_2)]\text{Cu}(\text{L})$ (where $\text{L} = \text{C}_2\text{H}_4$, *cis*- C_4H_8 , CHCH_3 , CHCH_3 , and CO) have been prepared from $[(\text{Ph}_3\text{B})\text{CH}(3,5\text{-(CH}_3)_2\text{Pz}_2)]\text{Li}(\text{THF})$, copper(I) triflate, and the corresponding coligand. These complexes have been characterized by NMR spectroscopy and X-ray crystallography. In all cases the bis(pyrazolyl) moiety is bound in $\kappa^2\text{N}$ fashion with the BPh_3 group rotated to sit over the metal center, sometimes coordinating to the metal via phenyl carbons as in $[(\text{Ph}_3\text{B})\text{CH}(3,5\text{-(CH}_3)_2\text{Pz}_2)]\text{Li}(\text{THF})$ and $[(\text{Ph}_3\text{B})\text{CH}(3,5\text{-(CH}_3)_2\text{Pz}_2)]\text{Cu}(\text{CO})$ or simply hovering above the metal site as in $[(\text{Ph}_3\text{B})\text{CH}(3,5\text{-(CH}_3)_2\text{Pz}_2)]\text{Cu}(\text{C}_2\text{H}_4)$ and $[(\text{Ph}_3\text{B})\text{CH}(3,5\text{-(CH}_3)_2\text{Pz}_2)]\text{Cu}(\text{cis-CH}_3\text{CH=CHCH}_3)$. The ^{13}C and ^1H resonances of the ethylene carbon and protons of $[(\text{Ph}_3\text{B})\text{CH}(3,5\text{-(CH}_3)_2\text{Pz}_2)]\text{Cu}(\text{C}_2\text{H}_4)$ appear at δ 81.0 and 3.71 ppm in CD_2Cl_2 , respectively. The characteristic CO frequency for $[(\text{Ph}_3\text{B})\text{CH}(3,5\text{-(CH}_3)_2\text{Pz}_2)]\text{Cu}(\text{CO})$ has been observed at $\bar{\nu}$ 2092 cm^{-1} by infrared spectroscopy and is lower than that of free CO suggesting moderate $\text{M} \rightarrow \text{CO} \pi$ -back-donation. A detailed analysis of these complexes has been presented herein.



INTRODUCTION

Poly(pyrazolyl)borates have been widely explored by inorganic, organometallic, and coordination chemists since their discovery in 1966.^{1–6} These monoanionic nitrogen-based ligands are particularly attractive due to the ability to fine-tune their steric and electronic properties and thereby modulate the reactivity of the bound metal center by changing the number and nature of the substituents on the pyrazolyl moieties and on the boron. The bulk of the attention in these ligands has been paid to

monoanionic, bis(pyrazolyl)methylborate ligands allow the synthesis of neutral metal adducts with $\text{M}(\text{I})$ salts including those of copper(I). In particular, we describe the preparation of $[(\text{Ph}_3\text{B})\text{CH}(3,5\text{-(CH}_3)_2\text{Pz}_2)]^-$ (Figure 1) and its utility in the synthesis of $[(\text{Ph}_3\text{B})\text{CH}(3,5\text{-(CH}_3)_2\text{Pz}_2)]\text{CuL}$ complexes containing $\text{L} = \text{ethylene}$, carbon monoxide, and *cis*-2-butene. This bis(pyrazolyl)methylborate ligand does not have the sometimes problematic and reactive B–H moiety found in common bis(pyrazolyl)borates.^{11,19} It features a negatively charged boron that is significantly less prone to coordination

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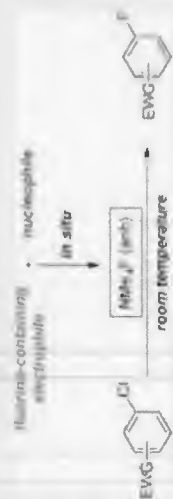
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Multiple Approaches to the In Situ Generation of Anhydrous Tetraalkylammonium Fluoride Salts for S_NAr Fluorination ReactionsMegan A. Cismesia,[†] Sarah J. Ryan,[†] Douglas C. Bland,[‡] and Melanie S. Sanford^{*,†}[†]Department of Chemistry, University of Michigan, 930 North University Avenue, Ann Arbor, Michigan 48109, United States[‡]Core Research & Development, The Dow Chemical Company, 1710 Building, Midland, Michigan 48674, United States

Supporting Information

ABSTRACT: This article focuses on the development of practical approaches to the in situ generation of anhydrous fluoride salts for applications in nucleophilic aromatic substitution (S_NAr) reactions. We report herein that a variety of combinations of inexpensive nucleophiles (e.g., tetraalkylammonium cyanide and phenoxide salts) and fluorine-containing electrophiles (e.g., acid fluoride, fluorosulfonate, benzenesulfonyl fluoride, and aryl fluorosulfonate derivatives) are effective for this transformation. Ultimately, we demonstrate that the combination of tetramethylammonium 2,6-dimethylphenoxide and sulfonyl fluoride (SO_2F_2) serves as a particularly practical route to anhydrous tetramethylammonium fluoride. This procedure is applied to the S_NAr fluorination of a range of electron-deficient aryl and heteroaryl chlorides as well as nitroarenes.



INTRODUCTION

Fluorinated arenes and heteroarenes appear in a variety of pharmaceuticals and agrochemicals.¹ In many cases, the replacement of a C–H bond with a C–F bond is used to increase the bioavailability, metabolic stability, and/or lipophilicity of these biologically active molecules.² However, despite their prevalence, many aryl and heteroaryl fluorides remain challenging to synthesize, particularly under mild conditions and with inexpensive reagents.

One of the most common methods for the formation of aryl fluorides is nucleophilic aromatic substitution (S_NAr fluorination). These transformations typically involve the reaction of an aryl chloride or nitroarene with an alkali metal fluoride salt (e.g., NaF, CsF, or TBAF) or a fluoride source (e.g., SO_2F_2) and base.

temperature. This reagent proved highly reactive for the S_NAr fluorination of electron-deficient aryl chlorides and nitroarenes.^{3a–f} However, widespread application of this method remains limited by both cost (C_6F_6) and toxicity (NBu_4CN) considerations.

Our overall objective is to develop more practical and potentially scalable approaches to the in situ generation of anhydrous fluoride salts for S_NAr reactions (Scheme 1c). We report herein that a variety of combinations of inexpensive nucleophiles and fluorine-containing electrophiles are effective for this transformation. Ultimately, we demonstrate that the reaction of tetramethylammonium 2,6-dimethylphenoxide with sulfonyl fluoride (SO_2F_2) serves as a particularly practical route to anhydrous tetramethylammonium fluoride (NMe_4F). This

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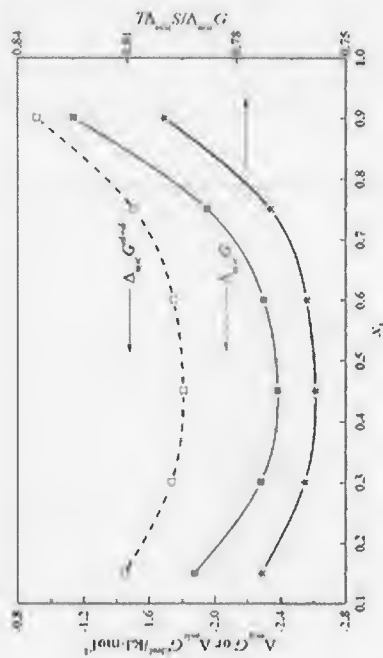
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Interaction and Micellar Behavior of Binary Mixture of Amino Sulfonate Amphiphilic Surfactant with Octadecyltrimethylammonium Bromide in Aqueous Solutions of NaCl

Zhao Hua Ren*, Jing Huang, Yan Cheng Zheng, Lu Lai, and Lin Li Hu

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JOURNAL OF combinatorial CHEMISTRY

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Volume 11, Number 6

November/December 2009

Report

Preparation and Optimization of Iridium Complexes with Quinazolinone Ligands on Solid Supports

Nai-Mu Hsu, Chung-Yen Li, Chun-Mei Yang,
Tzu-Sheng Lin, Bi-Han Hu, Yogesh S. Tingare,
Wei-Chun Chang, Gaurav Kumar Srivastava, and
Wen-Ren Li*

*Department of Chemistry, National Central University,
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Received July 18, 2009

Solid-phase combinatorial techniques are tremendously useful in the search for new drugs for the treatment of a wide variety of ailments.^{1–4} Materials discovery, however, has yet to benefit significantly from combinatorial methodology. Indeed, only a few exceptional attempts have been made to apply combinatorial approaches to the discovery of materials possessing novel photophysical properties.^{5–8} Moreover, even among these few reports, most of the

OLEDs are advanced alternatives to inorganic light-emitting diodes and liquid-crystal displays. Because of their excellent properties (flexibility, self-luminescence, rapid response, good contrast, low energy consumption), they hold great potential for application in flat-panel displays. In recent decades, much attention has been focused on organometallic complexes of various heavy metals, including Ir,^{19–26} Os,²⁷ Ru,^{28,29} and Pt,³⁰ for their potential use as phosphorescent dopants in OLEDs. Iridium complexes, in particular, are excellent emitters for OLED applications.^{19–26} In 2006, we used a combinatorial organometallic solid phase synthesis and high throughput screening methodology to identify a novel iridium complex having two cyclometallating ligands and a single monoanionic, bidentate ancillary ligand.³¹ The optimization of lead organometallic materials using this library approach, however, remains in its nascent stages.^{32–34} As part of a continuing effort toward the development of organometallic materials exhibiting desired properties, we are aiming to optimize the properties of organometallic complexes through structural modifications using solid phase synthesis. So far, the key ligands we have used have been

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Journal of Medicinal Chemistry

Perspective

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A Drug of Such Damned Nature.¹ Challenges and Opportunities in Translational Platinum Drug Research

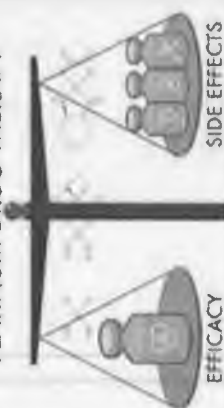
Miniperspective

Dorian M. Cheff and Matthew D. Hall*

NCATS Chemical Genomics Center, National Center for Advancing Translational Sciences, National Institutes of Health, 9800 Medical Center Drive, Rockville, Maryland 20850, United States

ABSTRACT: The platinum-based anticancer agents cisplatin, carboplatin, and oxaliplatin represent a spectacular translational science achievement. The basic research observations that led to the discovery of Pt complexes as DNA-binding agents that elicit cell arrest, the preclinical tumor regression studies, and the inorganic medicinal chemistry that led to clinical implementation of effective platinum complexes in the clinic have fueled multidisciplinary research into platinum-based drugs. While the successes are clear and the research activity continues, a significant window of time has passed since a new Pt drug has been approved for clinical use. Here we assess the current Pt drug landscape and challenges for future Pt development and discuss opportunities for improving our understanding of Pt drugs that utilize contemporary translational science tools such as chemical biology and real-time imaging. The underexplored spaces may reveal new opportunities for Pt drug development.

PLATINUM DRUG THERAPY



■ INTRODUCTION

Platinum stands out among chemotherapeutic classes for its spectacular successes and utility in combination therapy, but also for its inconvenience to the patient and the resistance that can arise. The first report related to the discovery of cisplatin was published over 50 years ago (1965) (see Hoeschele² for a detailed description of the early experiments). However, as is well reported, it was likely first synthesized by Michele Peyrone around 1840³ and was critical to Nobel prize-winner Alfred Werner's work on inorganic isomerism.⁴

Cisplatin became the first of three platinum-based anticancer

drug candidates, to characterize the ways in which cells become resistant to Pt drugs, to explore whether other (non-Pt) metal complexes can offer useful biological activity, to study the (bio)chemistry of Pt complexes in the context of reactions with biomolecules, and to characterize the cellular pharmacology and fate of Pt drugs.

This review aims to serve as a reflection on the ghosts of platinum research past, present, and future. It is written to focus attention and inspire discussion on the strengths of the field and the future of Pt research. It was inspired partly by Richard Callaghan's honest reflections on the relevance of drug

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JOURNAL OF NATURAL PRODUCTS

Editorial

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Celebration of Volume 75 of the *Journal of Natural Products*

The year 2012 marks the 75th issue of the *Journal of Natural Products* and its predecessor journal *Lloydia*. At its inception in 1938, *Lloydia* was a quarterly journal focusing primarily on mycology and plant anatomy. It became the official scientific organ of the American Society of Pharmacognosy (ASP) in 1961, when it began to have more of a chemical emphasis on natural products. The name was changed to the *Journal of Natural Products* in 1979, and the journal became a monthly publication in 1992, having been published bimonthly since 1975. In 1996, a significant change occurred when the journal became a copublication of the American Chemical Society and ASP. It is planned to celebrate the current milestone indicating the longevity of this publication, in a symposium dedicated to the *Journal of Natural Products* at the forthcoming International Congress on Natural Products Research to be held in New York City during the period July 28 to August 1, 2012. Three distinguished natural product scientists who are frequent contributors to the journal will participate in this symposium.

The March 2012 issue of the *Journal of Natural Products* will be dedicated to Dr. Gordon M. Cragg, formerly Chief, Natural Products Branch, National Cancer Institute (NCI), Frederick, Maryland. In addition to his past work in developing new anticancer drugs from natural product sources, Dr. Cragg is very well known for his great interest in the international development of natural products research. Drs. Vanderlan Silva da Bolzani, Michael T. Davies-Coleman, David J. Newman, and Shiao B. Singh are serving as Guest Editors for this special issue.

This year, the first of two journal cover illustrations to be used shows a grouping of marine and terrestrial organisms from which either marketed anticancer drugs or antineoplastic agents currently in phase clinical trials have been obtained, either in their natural form or as semisynthetic derivatives, and for which the relevant structures are overlaid. This was provided by Dr. David J. Newman, of NCI, Frederick, Maryland. The cover illustration for the second half of 2012 will feature a motif to be

and their coauthors! These papers and those of all past winners of the Schwaning and Beal Awards may be readily accessed on the journal Web page (<http://pubs.acs.org/jnp>).

A. Douglas Kinghorn, Editor-in-Chief



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Daryl Good, Charlie Pham, Jacob Jagas, József R. Lewandowski, and Vladimir Ladizhansky

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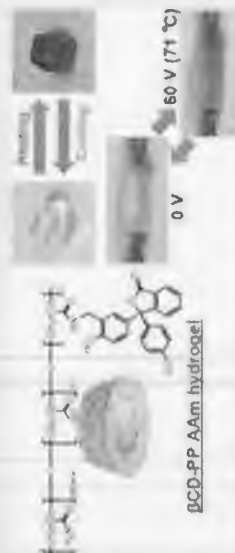
Multifunctional Stimuli-Responsive Supramolecular Materials with Stretching, Coloring, and Self-Healing Properties Functionalized via Host–Guest Interactions

Yoshinori Takashima,[†] Koki Yonekura,[†] Kohei Koyanagi,[†] Kazuhisa Iwaso,[†] Masaki Nakahata,[†] Hiroyasu Yamaguchi,[†] and Akira Harada^{*,†,‡}

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Supporting Information

ABSTRACT: The visualization of changes in the stress and bonding state inside polymeric materials is an attractive function in materials science. In this study, phenolphthalein (PP) was selected to prepare stimuli-responsive coloring materials. PP becomes purple under basic conditions in aqueous solutions; however, PP becomes colorless under basic conditions when it forms a complex with β -cyclodextrin (β CD). To exploit this property of PP, we prepared a color-changing hydrogel (β CD-PP AAm hydrogel) based on acrylamide (AAm) as the main chain and β CD and PP moieties as the side chains. The β CD-PP AAm hydrogel exhibits a color change when heat or a competing molecule is applied at a pH less than 8. This color change was confirmed by ultraviolet–visible (UV–vis) spectroscopy, and the mechanical properties were determined via compression and tensile measurements. The β CD-PP AAm hydrogel also exhibits a rapid, reversible color change upon Joule heating produced by an electric current passing through the gel.



1. INTRODUCTION

Recently, stimuli-responsive and functional polymeric materials have become emerging areas of research.^{1–6} Representative functions of such materials include self-healing,^{7–13} actuation,^{14–32} sensors,^{33–39} and drug delivery.^{40–46} However, fabricating polymeric materials with stimuli responsiveness or multiple functions, such as biological materials based on simple molecular design, remains difficult

relatively easy to endow with functions through the association and dissociation of an inclusion complex with CDs and guest molecules. Herein, we selected phenolphthalein (PP)^{49,50} as a guest molecule because an aqueous solution of an inclusion complex between β CD and PP does not exhibit a purple color at a pH of 10. However, the addition of competitive guest molecules colors the solution (Figure 1a).^{71,72} Ritter and his co-workers studied the interaction of β CD derivatives with pH-

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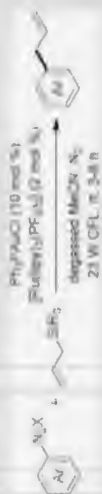
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Cross-Coupling Reactions of Aryldiazonium Salts with Allylsilanes under Merged Gold/Visible-Light Photoredox Catalysis

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Supporting Information

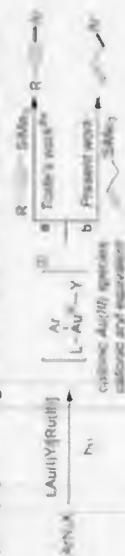
ABSTRACT: A method for the cross-coupling reactions of aryl diazonium salts with triallylsilanes via merged gold/photoredox catalysis is described. The reaction is proposed to proceed through a photoredox-promoted generation of an electrophilic arylgold(III) intermediate that undergoes transmetalation with allyltrimethylsilane to form allylarenes.



Homogeneous gold catalysis has received a great deal of attention over the past decade. In the majority of these transformations, the π -acidity of gold complexes triggers the activation of C–C multiple bonds such as alkenes, allenes, and alkynes, thereby favoring the attack of nucleophile.¹ Unlike other late transition metals, gold(I) complexes are generally unreactive toward the oxidative addition of aryl and vinyl halides and pseudohalides, limiting the potential application of gold in the field of coupling reactions.² Recent research revealed that the realization of reactivity similar to that of late transition metals is possible with two-electron redox processes wherein Au(III) intermediates are accessible.³ However, those reactions utilize sacrificial oxidants such as I^+ derivatives or F^+ sources and often require harsh reaction conditions.⁴

Recently, Glorius and co-workers have pioneered an overall redox-neutral strategy for accessing Au(III) intermediates using merged gold/photoredox catalysis, effecting oxyarylation reaction of alkenes.⁵ Since then, the merged gold/photoredox

Scheme 1. Cross-Coupling Reaction of Aryl Diazonium Salts $[\text{ArN}_2^+\text{X}^-]$ with Organoallylsilanes: Known and Present Work



Allylarenes represent an important class of substituted aromatic compounds¹¹ which are traditionally accessed via (1) Friedel–Crafts allylation,¹² (2) reaction of arylmetals (magnesium, copper etc.) with allylic halides¹³ or with π -allyl palladium complexes,¹⁴ and (3) palladium/copper-catalyzed reaction of aryl halides with allylmetals (magnesium, copper etc.), allylboronic acids, allylstannanes, or allylsilanes.¹⁵ Most of these approaches often require fluoride/base sources, sophisticated ligand systems, or harsh reaction conditions. Among other approaches, the work of Albini and co-workers concerning the photosensitized decomposition of benzene-

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