

From Academia to the Market – Air-stable Ni(II)/Josiphos Catalysts

Florian Bächle^a, Achim Link^{*a}, Abderrahmane Amgoune^b, and Anis Tlili^{*b}

Abstract: The design, synthesis, commercialization and application of air-stable Ni(II)/Josiphos complexes has been realized in a collaboration between Solvias and ICBMS (University Lyon 1). The Ni-complexes are utilized as versatile precatalysts for diverse cross-coupling reactions. Apart from being active in established C–C and C–N couplings at low catalyst loadings, the novel Ni-precatalysts enabled the development of the challenging monoarylation of ammonia, ammonia surrogates and even alkylammonium chlorides with aryl carbamates. Finally, the α -arylation of acetone with aryl chlorides, carbamates and pivalates was demonstrated using the Ni(II)/Josiphos precatalysts.

Keywords: Acetone · Ammonia · Catalysis · Josiphos · Nickel



Florian Bächle studied chemistry at the University of Heidelberg and received his diploma in organic chemistry in the group of Prof. Dr. G. Helmchen. Subsequently, he joined the research group of Prof. Dr. A. Pfaltz for his PhD thesis at the University of Basel. He started his industrial career as Lab head in process development. In March 2018, he joined Solvias taking over the responsibility for the Ligand and Catalysis

business as Leading Scientist and Product Manager.



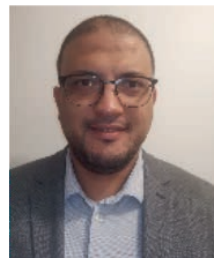
Achim Link received his MSc degree (2013) in the group of Prof. C. Sparr at the University of Basel and was involved in launching the group's first research project. He remained for his PhD studies focusing on bifunctional organometallic reagents. Achim received a fellowship from the Camille & Henry Dreyfus Foundation and an Early Postdoc Mobility fellowship from the Swiss National Science Foundation

allowed him to join Prof. S. L. Buchwald at MIT investigating CuH-catalyzed reactions. In 2020, he joined Solvias as a project leader in the catalysis group. His research interests range from homogeneous asymmetric hydrogenations and hydrofunctionalizations catalyzed by earth abundant metals to cross coupling reactions and photocatalytic transformations.



Abderrahmane Amgoune received his PhD in 2006 at the University of Rennes (Profs. J.-F. Carpentier and C. Thomas), France. He then moved to the University of Konstanz as an Alexander-von-Humboldt postdoctoral research associate with Prof. S. Mecking. In 2008, he was appointed a CNRS research position at the University of Toulouse. In 2017, he was promoted to

full Professor at the University of Lyon (France). His research interests range from fundamental organometallic chemistry to the development of dual catalytic strategies with transition metals for the functionalization of inert bonds. His work has received several awards, including most recently a nomination as junior member at the Institut Universitaire de France (2019), the Thieme Chemistry Journals Award (2020) and the Young investigator Award from the French Chemical Society, Organic Chemistry division (2016).



Anis Tlili is CNRS Research Fellow. After studying chemistry in the Université de Bourgogne, Anis gained a master's degree in the same university with Prof. S. Jugé (Dijon, France). Afterwards, he completed his PhD in the group of Dr. M. Taillefer (Montpellier, France) in late 2011. Subsequently, he joined the group of Prof. M. Beller (LIKAT, Rostock Germany) for a postdoctoral stay before taking up a second

collaborative postdoctoral position (CEA/ICSN, Paris). In 2014, he was appointed a CNRS research Fellow in Lyon. His current research interests lie in the area of homogeneous catalysis/photoredox catalysis, with particular focus on fluorine chemistry. Anis was awarded recently a JSP Fellow for the 53rd Bürgenstock and was selected to participate to EuChemS young investigators workshop 2018 in Oxford.

1. Introduction

Since more than two decades, the Ligands & Specialty Products unit at Solvias provides full service in the field of homogeneous and heterogeneous metal catalysis. In addition, Solvias is a producer of ligands and catalysts for homogeneous catalyzed reactions from gram to multi-kilogram scale. The combination of a broad ligand and catalyst portfolio with capabilities in the development of chemocatalyzed reactions supported by a high-throughput experimentation (HTE) platform allows for the investigation of a wide variety of different transformations such as homogeneous and heterogeneous hydrogenations, C–C and C–X couplings, carbonylations, aminations and recently also

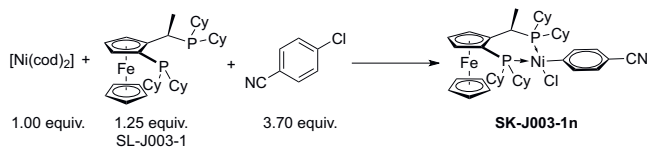
Correspondence: Dr. A. Link, E-mail: achim.link@solvias.com; Dr. A. Tlili*, E-mail: anis.tlili@univ-lyon1.fr.

^aSolvias AG, Römerpark 2, CH-4303 Kaiseraugst, Switzerland; ^bInstitute of Chemistry and Biochemistry (ICBMS–UMR CNRS 5246), Univ Lyon, Université Lyon 1, CNRS, CPE-Lyon, INSA, 43 Bd du 11 Novembre 1918, 69622 Villeurbanne, France

transformations enabled by photoredox-catalysis. With a strong focus on applying latest scientific findings in the development of commercial catalytic processes, various collaborations with academia were established.^[1] Amongst others, a long-standing co-operation with the University of Basel which was started in 2007 with the commercialization of iridium-UBAPHOX complexes for asymmetric hydrogenation. More recently, a new collaboration in the field of photocatalysis was launched with Prof. Sparr to introduce a novel organic aminoacridinium photocatalyst to the market.^[2] Another key technology of interest towards sustainable chemistry is the application of non-precious metals in modern catalysis. In 2015, a collaboration on Ni-catalyzed reactions was started between the ICBMS (University of Lyon) and Solvias, which is followed-up by a new and exciting internal program on Ni-catalyzed aminations. Nickel is a highly attractive transition metal. Besides its high availability and low cost, its reactivity especially enabling facile oxidative addition of aryl chlorides or aryl ethers is of prime interest. Moreover, radical pathways are well-accessible with Ni, while β -hydride elimination tends to be slower in comparison to Pd. These properties led to the advent of 'Ni-photoredox dual catalysis' as a new tool to create challenging bonds under comparatively mild conditions. A main drawback in Ni-chemistry is the high air and moisture sensitivity of Ni(0) precatalysts such as Ni(COD)₂. In this context, we launched a research program to investigate the synthesis as well as the application of air stable Ni(II)/Josiphos precatalysts, of which several have been commercialized. These precatalysts allowed to achieve the challenging arylation of primary building blocks such as ammonia and acetone. Independently, Cornella as well as Engle recently developed two air-stable and now commercially available Ni(0)-precursors: Ni(4-^tBu₃stb)₃ and Ni(COD)(DQ).^[3]

2. Synthesis of Air-stable Nickel (II) catalyst

The synthesis of a new air- and moisture-stable nickel (II)/Josiphos precatalyst has been developed. Treating 4-chlorobenzonitrile with substoichiometric amounts of Ni(COD)₂ and **SL-J003-1** allowed to isolate the desired stable Ni(II)-precatalyst **SK-J003-1n** (Scheme 1).^[4] Herein, the nitrile-group has a dual role. Its electron-withdrawing character facilitates the reductive elimination to set free the desired reactive L-Ni(0) species while at the same time forming benzonitrile derivatives in catalytic amounts in the reaction media. These latter species could further enhance the stability of Ni(0) intermediate by forming Ni(η^2 -NC-Ar) complexes as demonstrated earlier by the Hartwig group.^[5] The same synthetic protocol can easily be extended to other Josiphos ligands enabling the formation of a plethora of air-stable, but in reaction media highly active, Ni(II)-precatalysts.

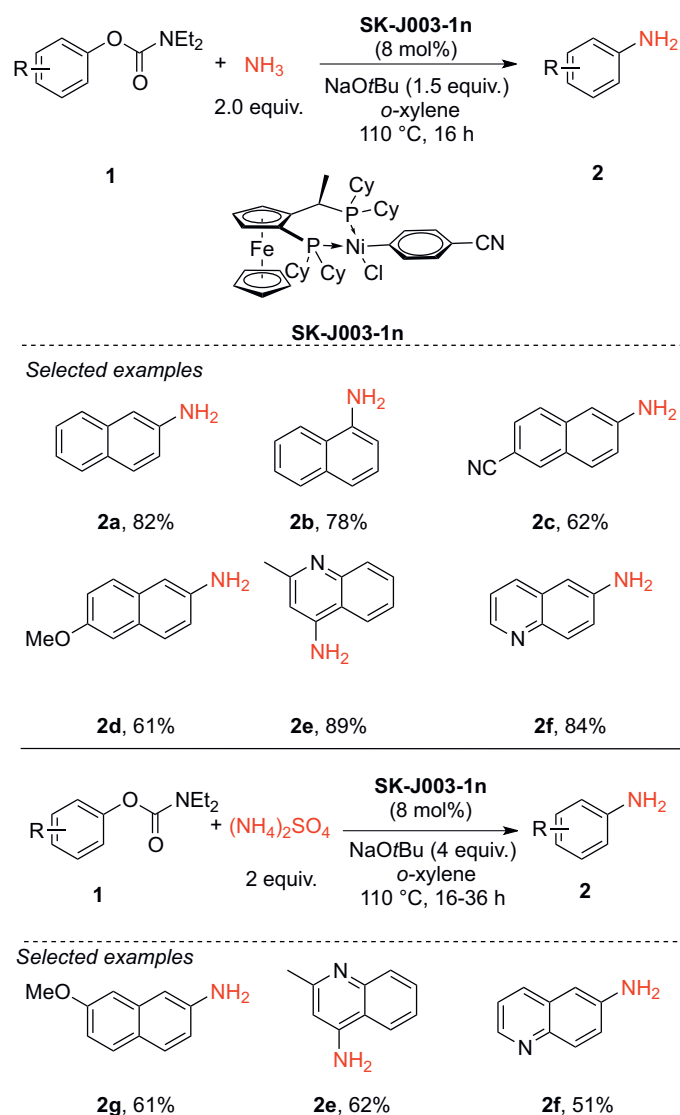


Scheme 1. Synthesis of air-stable Ni/Josiphos precatalyst complex **SK-J003-1n**.

3. Nickel/Josiphos-catalyzed Arylation of Ammonia and Alkylamines Using Aryl Carbamates

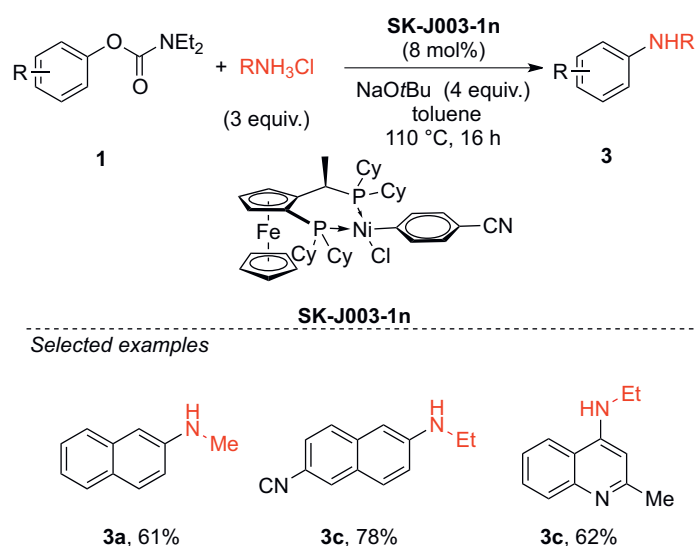
Aryl amines are ubiquitous intermediates and are widely employed for the manufacture of agrochemicals, dyes as well as pharmaceuticals.^[6] The direct access to aniline derivatives through mono selective arylation of ammonia has been considered as a huge challenge for a long time. Previous methods relied on the use of ammonia surrogates that increased costs and made it necessary to implement an additional synthetic step for deprotection,

that could lower the functional group tolerance in complex molecules. To avoid these drawbacks, the challenging mono arylation of ammonia has been addressed under palladium catalysis^[7] by the groups of Hartwig,^[8] Buchwald,^[9] Beller^[10] and Stradiotto.^[11] Another important breakthrough was recently achieved by the groups of Hartwig^[4] and Stradiotto^[12] by employing Ni/Josiphos catalysts for the arylation of ammonia starting with aryl chlorides and tosylates. As a new step forward, we investigated the use of air-stable nickel(II) precatalysts for the arylation of ammonia with aryl carbamate derivatives as substrates (Scheme 2). The reaction was performed using a solution of ammonia in dioxane (0.5 M) in the presence of NaOtBu as base in *o*-xylene as solvent at 110 °C for 16 h.^[3] Naphthalene carbamate derivatives substituted with electron-withdrawing or electron-donating groups were tolerated and the desired anilines derivatives were obtained in good to excellent yield. Interestingly, heterocyclic arylcarbamates could also be used as starting material under the identified reaction conditions. Noteworthy, aniline products could also be obtained by using ammonium sulfate as an ammonia surrogate. Here, a greater excess of NaOtBu was required to reach similar reaction outcomes compared to the procedure using ammonia. It should be mentioned, that in the meantime, the Stradiotto group also developed a similar strategy for the arylation of ammonia using aryl carbamates, sulfamates and pivalate derivatives.^[13]



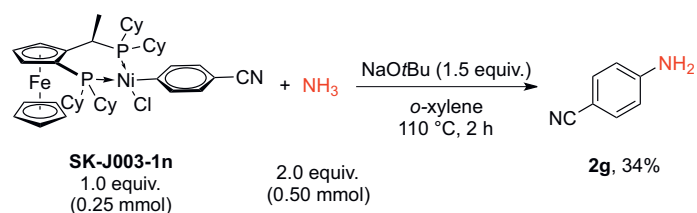
Scheme 2. Nickel-catalyzed amination of aryl carbamates with ammonia or ammonium sulfate.

In the following, we also investigated the challenging arylation of methyl- and ethylamines. In this context, the corresponding methyl and ethyl ammonium chloride salts were used as coupling partner along with aryl carbamate under the same system reported for the arylation of ammonia except for the excess of NaOtBu (4 equiv.) and the use of toluene as solvent. The desired products were obtained with good selectivity (Scheme 3).



Scheme 3. Nickel-catalyzed amination of aryl carbamates with alkyl ammonium chlorides.

In order to get more insight into the reaction mechanism, the air-stable Ni(II) catalyst **SK-J003-1n** was treated with ammonia (2 equiv.) in the presence of NaOtBu (1.5 equiv.). The desired product **2g** was obtained in 34% yield within 2 hours reaction time at 110 °C (Scheme 4). Preliminary ^{31}P NMR studies indicate the formation of Ni($\eta^2\text{-NC-Ph-4NH}_2$). Thus, this result is consistent with a Ni⁰/Ni^{II} catalytic cycle.

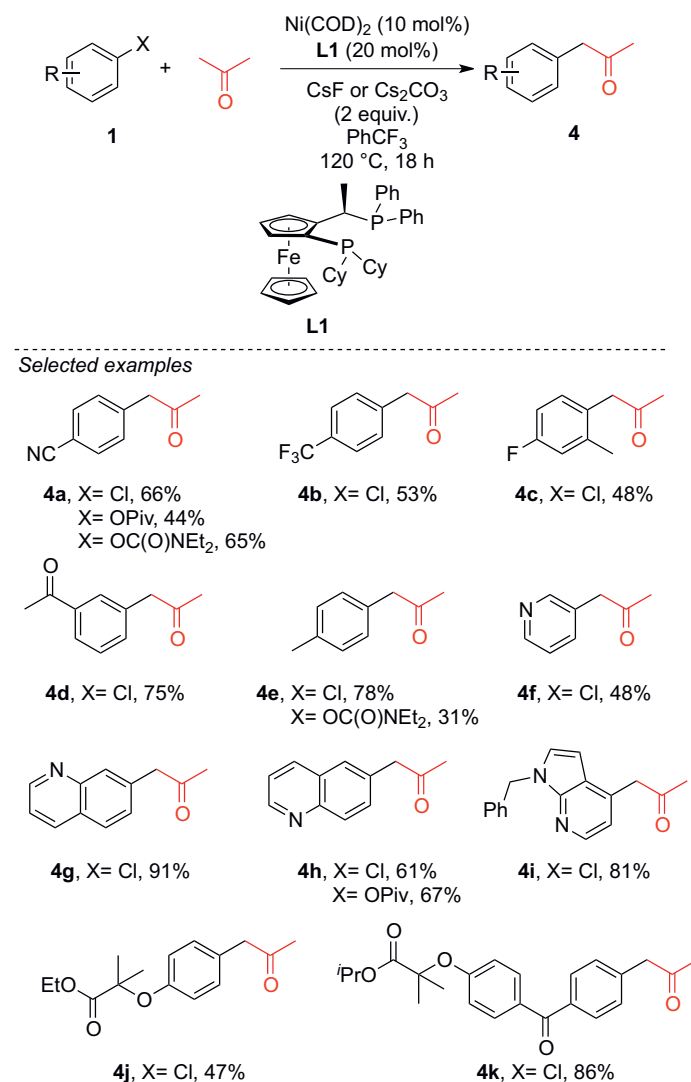


Scheme 4. Stoichiometric reaction of **SK-J003-1n** with ammonia.

2.1 Nickel/Josiphos-catalyzed α -Arylation of Acetone

The direct α -arylation of substrates with activated (acidic) C–H bonds under transition metal catalysis is a powerful tool to access compounds possessing a benzylic carbonyl moiety.^[14] In this context, several procedures have been developed since the pioneering work of Miura, Buchwald and Hartwig.^[15] Similar to the arylation of ammonia, the mono selective α -arylation of acetone turned out to be a challenging task as the mono-arylated acetone is prone to undergo further arylation. This challenge has been addressed by the group of Stradiotto in 2011 under palladium catalysis.^[16] Afterwards, several methodologies have been developed using palladium employing diverse ancillary ligands.^[17] We envisioned to develop the nickel-catalyzed α -arylation of acetone by applying the nickel/Josiphos system as alternative to well-established palladium processes. After optimization, it turns out that the as-

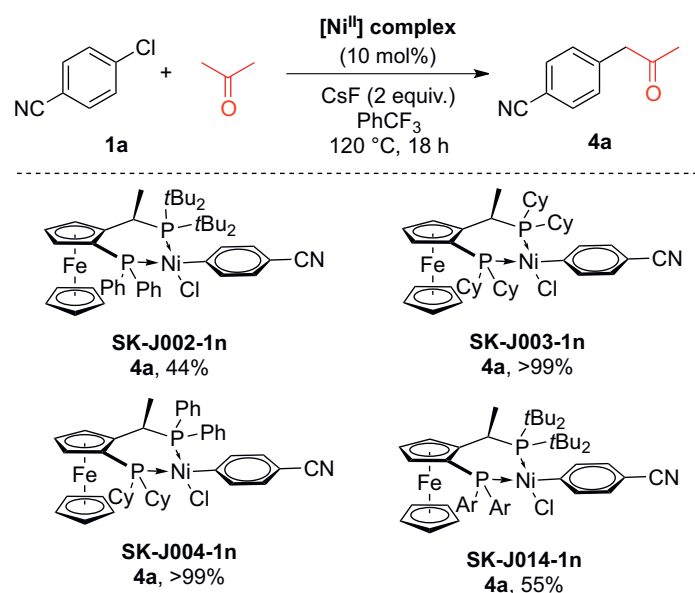
sociation of Ni(COD)₂/Josiphos **L1** allows the mono-selective α -arylation of acetone using aryl chlorides, aryl pivalates as well as aryl carbamates derivatives. The reactions were performed in the presence of CsF or Cs₂CO₃ as base in trifluorotoluene as solvent at 120 °C (Scheme 5). The reactions tolerate the presence of both electron-withdrawing and electron-donating groups. In addition, heterocyclic compounds were tolerated under the reaction conditions and the desired products were obtained in good to excellent yields. Also more complex aryl chloride starting materials could be converted to the desired product in moderate to excellent yield (products **4j** & **4k**, Scheme 5).^[18]



Scheme 5. Nickel-catalyzed α -arylation of aryl chlorides, carbamates and pivalates.

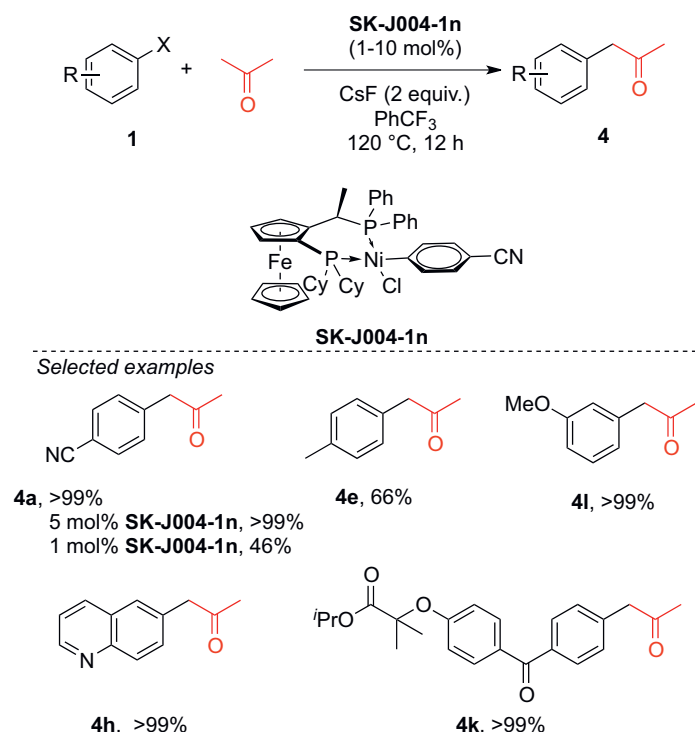
Next, we decided to investigate the use of air/moisture stable Ni(II) catalysts for the α -arylation of acetone. The reactions were performed in the presence of 10 mol% of Ni(II) precursor. It turns out that with **SK-J002-1n**, desired compound **4a** was afforded in only 44% yield. However, **SK-J003-1n** and **SK-J004-1n** allowed the full conversion of the starting material and in both cases the desired product **4a** was obtained in an excellent yield of >99%. In contrast, a diminished yield of 55% was obtained when using precatalyst **SK-J014-1n** (Scheme 6).

With these results in hand, we decided to investigate the reaction scope with the air-stable **SK-J004-1n** (Scheme 7). Remarkably, the same excellent yield for compound **4a** was obtained when



Scheme 6. Air stable nickel(II) complexes for the α -arylation of acetone with 4-chlorobenzonitrile.

5 mol% of precatalyst was employed. Even a lower catalyst loading of 1 mol% still delivered the product in an acceptable yield of 46%. Although a good yield of 66% of the desired product **4e** was obtained, a slight decrease was observed in comparison to the use of Ni(0) precatalyst and an excess of **L1**. Full conversions and excellent yields of >99% were obtained for compounds **4l**, **4h** and **4k**. It should be mentioned that the air-stable Ni(II) precatalyst afforded higher conversion and selectivity for product **4h**.



Scheme 7. α -Arylation of chloroarenes with the air-stable **SK-J004-1n**.

Mechanistic investigations, including stoichiometric reactions, isolation and full characterization of key intermediates in the solid state and in solution suggest a Ni⁰/Ni^{II} cycle. Interestingly, comparative analysis of a series of ligands, indicated that Josiphos-

type ligands play a key role in the stabilization and catalytic reactivity of Ni(II) intermediates. Moreover, the presence of an excess of **L1** allowed the formation of the (L1)₂Ni(0) intermediate, which was demonstrated to be catalytically active. Thus, the presence of an excess of ligand may enhance the stability of the present Ni(0) species. Regarding Ni(II) precatalyst, the presence of benzonitrile in the media may also improve the stability of Ni(0) through η^2 -coordination of the cyano group to the metal center.

5. Conclusion

We demonstrated that the combination Ni/Josiphos is an efficient catalytic system for the arylation of primary building blocks including ammonia and acetone. The catalytic system is highly efficient towards the activation of aryl chlorides, carbamates and pivalates substrates. Furthermore, commercially available and air-stable Ni(II) precatalysts are also highly efficient and easier to handle in comparison with very sensitive and difficult to handle Ni(0) precursors. Moreover, the presence of benzonitrile in the starting Ni(II) catalyst could be beneficial for the stabilization of the active Ni(0) catalyst. Josiphos ligands have shown once again their particular efficiency especially for the α -arylation of acetone since no other known phosphine or carbene ligands tested under the developed conditions have shown reactivity. The Solvias/ICBMS (University of Lyon 1) partnership is a fruitful industrial/academia collaboration with already major discoveries in the field of nickel-catalyzed cross-coupling processes. The application of Ni/Josiphos catalysts is still under joint investigation for the development of new and original methodologies.

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