

Tetrabutylammonium Iodide¹

$n\text{-Bu}_4\text{NI}$

[311-28-4] $\text{C}_{16}\text{H}_{36}\text{IN}$ (MW 369.43)
 InChI = 1/C16H36N.HI/c1-5-9-13-17(14-10-6-2,15-11-7-3)16-12-8-4;/h5-16H2,1-4H3;1H/q+1;/p-1/fC16H36N.I/h;1h/qm;-1
 InChIKey = DPKBAXPHAYBPRL-ZKCUATCLCI

(phase-transfer catalyst;¹ ether cleavage;¹ alkyl iodide formation;¹ salt effects¹)

Physical Data: mp 148 °C.

Solubility: sol EtOH, H₂O.

Form Supplied in: anhydrous white solid.

Analysis of Reagent Purity: tetraalkylammonium salts can be titrated with potassium 3,5-di-*t*-butyl-2-hydroxybenzenesulfonate and iron(III) chloride.^{1a}

Purification: crystallize from toluene–petroleum ether, CH₂Cl₂–petroleum ether (or hexane), acetone, ethyl acetate, EtOH–ethyl ether, aq EtOH, or H₂O. The solid is then dried at 90 °C under high vacuum for 2 d.²

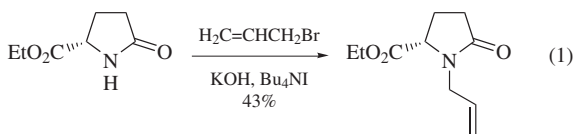
Handling, Storage, and Precautions: highly hygroscopic solid.

Original Commentary

André B. Charette

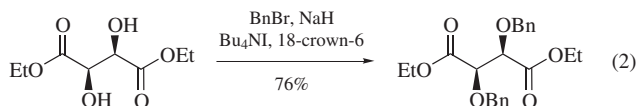
Université de Montréal, Montréal, Québec, Canada

Phase-transfer Catalyst. Although Bu₄NI is not as widely used as **Tetrabutylammonium Bromide**, it is often used in phase-transfer reactions. It was shown to promote several *N*-alkylation reactions³ of heterocycles such as pyrroles⁴ and of amides^{5,6} (eq 1).



It has also been used in phase-transfer carbene-mediated cyclopropanation reactions,⁷ metal-catalyzed carbonylation reactions of organic halides,^{8,9} and Wittig alkenation reactions.¹⁰

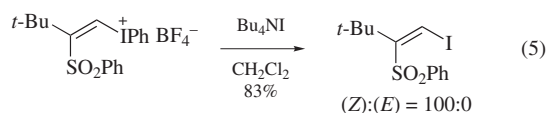
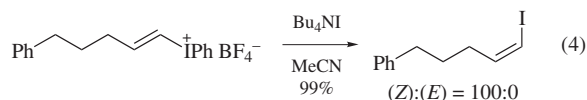
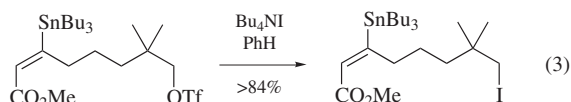
Several *O*-alkylation reactions¹¹ were shown to proceed much faster if Bu₄NI was added to the reaction mixture. One particularly interesting case is the benzylation of diethyl tartrate in which addition of a catalytic amount of Bu₄NI was shown to be essential for obtaining high yield of the desired product (eq 2).¹²



A mixture of **Zinc Chloride** and Bu₄NI catalyzes the synthesis of cyclic carbonates from oxiranes and carbon dioxide at rt and atmospheric pressure.¹³

Ether Cleavage. Methyl, allyl, and benzyl ethers derived from alcohols and phenols are regioselectively cleaved by a mixture of Bu₄NI and a Lewis acid (**Boron Trifluoride Etherate**¹⁴ or **Aluminum Iodide**¹⁵).

Alkyl and Vinyl Iodide. Bu₄NI can also be used as a source of iodide for nucleophilic displacement reactions. Neopentyl iodides can be prepared via S_N2 displacement of the corresponding triflate (eq 3).¹⁶ Alkenyliodonium tetrafluoroborates can be converted to vinyl iodides upon treatment with Bu₄NI (eqs 4 and 5).¹⁷



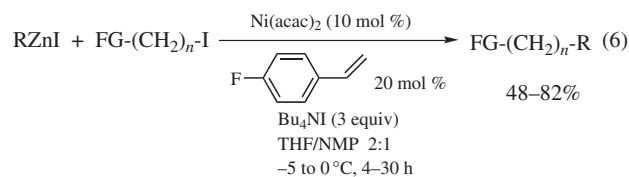
First Update

Petr Sazonov & Irina Beletskaya

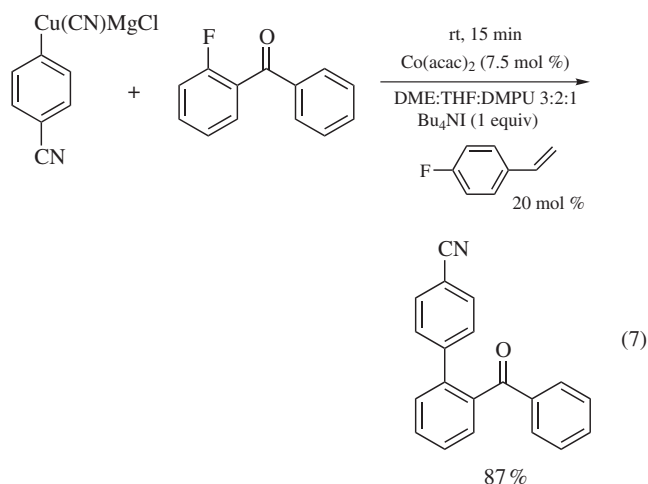
Moscow State University, Moscow, Russia

Transition-metal-catalyzed Cross Coupling. Bu₄NI (though less frequently than Bu₄NBr) is used as an additive or cocatalyst in cross-coupling reactions, often allowing use of less active aryl bromides, chlorides, or sulfonates instead of costly aryl iodides.

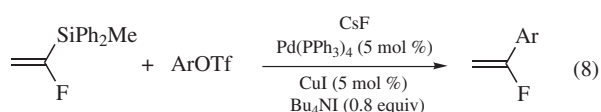
Bu₄NI was shown to accelerate and improve the selectivity of Ni-catalyzed cross coupling of various organozinc halides, including AlkZnHal, with aryl and alkenyl iodides, triflates, and nonaflates.^{18,19} Addition of 3 equiv of Bu₄NI (together with 4-fluorostyrene) was crucial in effecting the cross coupling of AlkZnHal with primary alkyl halides (eq 6).^{19,20}



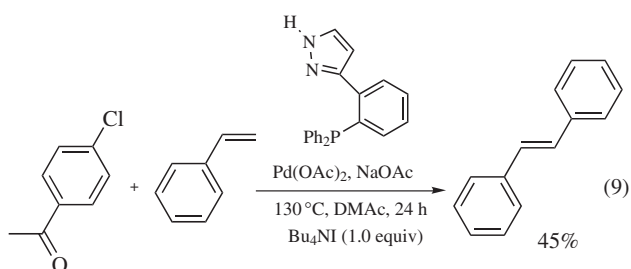
A similar effect of Bu₄NI (10 mol %) was found in the Cu^I-catalyzed reaction of Grignard reagents with benzylphosphonates.²¹ Addition of Bu₄NI also led to significant improvement in the reactions of polyfunctional arylcopper reagents with various ArX and HetX (X=Br, Cl, F, OTs) catalyzed by Co(acac)₂ (eq 7).^{22–24}



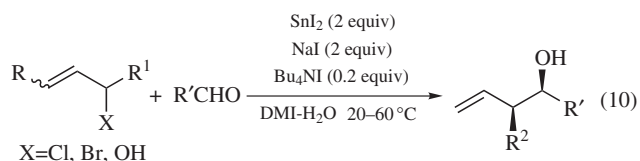
Bu₄NI proved to be an essential additive in the successful coupling of vinylsilanes and carbonylative coupling of vinylstannanes with aryl triflates catalyzed by Pd⁰ (eq 8).^{25,26}



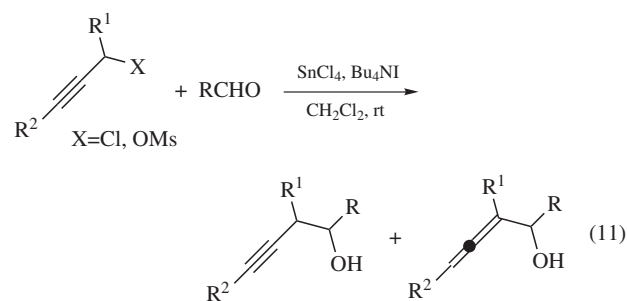
Bu₄NI increases the yield in the palladium-catalyzed carbonylation of aryl bromides²⁷ which was used in the synthesis of [carbonyl-¹¹C] amides.²⁸ The addition of Bu₄NI allowed Heck arylation of styrene with unactivated (palladacycle catalyst, 130 °C)²⁹ and activated (pyrazolylphosphine ligand) aryl chlorides (eq 9).³⁰



Alylation of Carbonyl Compounds and Related Reactions. SnHal₂/Bu₄NI-NaI (Hal=I, Br, Cl) system was used in γ -*syn*-diastereoselective Barbier-type allylation of aldehydes with allyl halides, alcohols, and mesylates (eq 10).^{31–33}

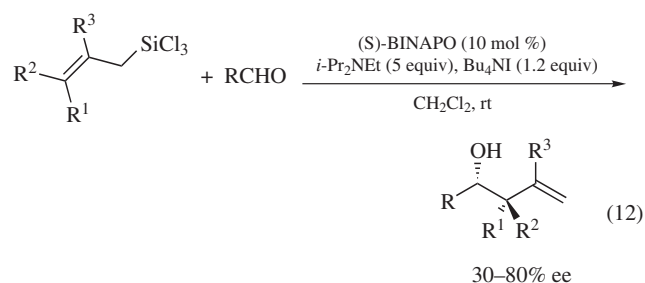


A similar reaction with propargyl halides or mesylates affords a mixture of propargylation and allenylation products.^{34,35} Tin^{IV} halides (Hal=Cl, I) can be used (in catalytic or stoichiometric amounts) instead of SnX₂, and the SnHal₄-Bu₄NI system is shown to be superior to SnI₂-Bu₄NI for the selective propargylation with 2-propynyl mesylates (eq 11).^{36–39}

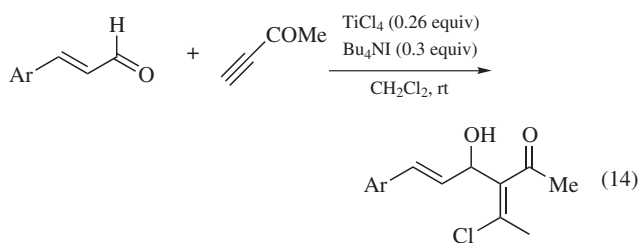
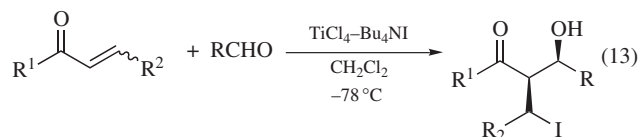


A SnI₄-Bu₄NI system was also employed for the Reformatsky-type condensation of α -bromoketones with aldehydes.⁴⁰

The allylation of aldehydes with allyltrichlorosilane in the presence of O-donor ligands, including its asymmetric version, is profoundly accelerated by Bu₄NI (eq 12). Bu₄NBr was found to be less effective.^{41,42}

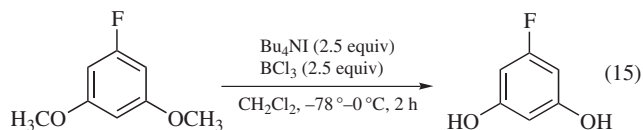


TiCl₄-promoted Baylis–Hillman Reaction. TBAI was found to be an important additive in the TiCl₄-promoted Baylis–Hillman reaction between α,β -unsaturated (vinylic, acetylenic, and cyclopropyl) carbonyl compounds and aldehydes.^{43–45} Bu₄NI works better than other Bu₄NX or Me₂S and can be sometimes used in catalytic amounts. Reaction is diastereoselective, and with alkenes usually gives *syn*-adducts (eqs 13 and 14).^{46–50}



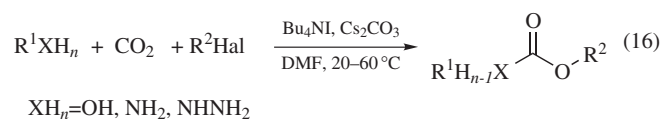
Ether and Ester Cleavage. Bu₄NI is a reagent for the dealkylation of alkyl sulfonates, sulfonium ions, and phosphonates. The dealkylation usually proceeds with quantitative yields and is often used in the synthesis of natural products.^{51–57} The combination of three reagents Bu₄NI, BF₃, and *N,O*-bis(trimethylsilyl) trifluoroacetamide was shown to be critical for the complete deethylation of CF₂P(O)(OEt)₂ groups in an oligopeptide.⁵⁸

A combination of Bu₄NI with BCl₃ is a generally applicable, selective and operationally simple reagent for the cleavage of primary alkyl aryl ethers at low temperatures (eq 15).^{59,60}



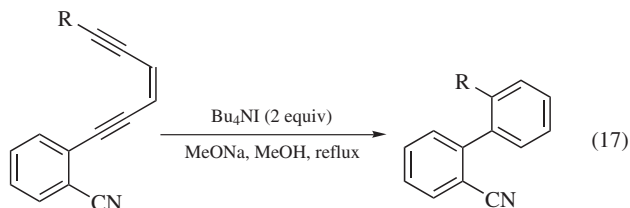
Catalytic Bu₄NI facilitates cyclic ether and lactone cleavage with 48% HBr under microwave irradiation conditions.⁶¹

Phase-transfer Catalysis. In the past decade Bu₄NI has continued to be used as a phase-transfer catalyst in the alkylation of O, N, C, S, Se, and P-centred nucleophiles.^{62–66} Of particular interest is its application in CO₂-fixation—the synthesis of mixed carbonates, carbamates, and carbazates by a three-component reaction of CO₂, alcohol(amine), and alkyl halide (eq 16).^{67–72}

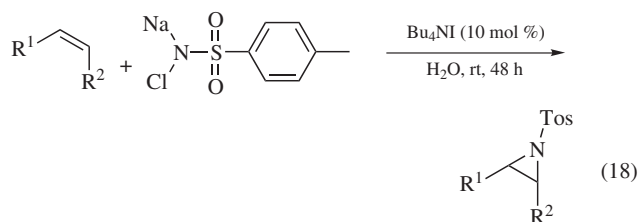


These conditions are also applicable to solid-phase synthesis.⁷³ The addition of Bu₄NI was essential in avoiding alkylation of amines in the synthesis of carbamates.⁷⁰ A similar system (Cs₂CO₃, Bu₄NI) was also employed for the synthesis of dithiocarbonates, dithiocarbamates, and phosphonodithioformates from CS₂ as well as for the N-alkylation of carbamates.^{74–77} In the synthesis of carbonates CS₂CO₃ can be replaced by K₂CO₃.^{78,79}

Using Bu₄NI as a phase-transfer catalyst allows control of the regioselectivity of [MeO][−]-promoted anionic cyclization of 1-aryl-3-hexen-1,5-diyne resulting in the selective formation of biaryl products (eq 17).⁸⁰

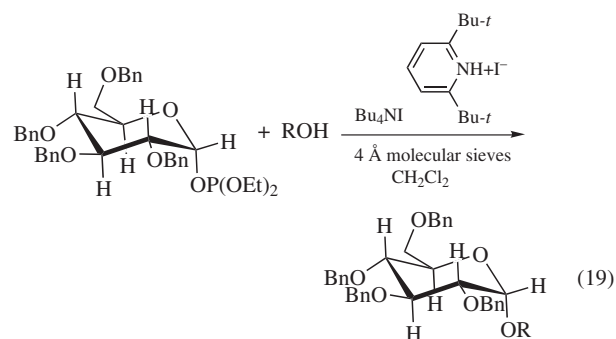


Miscellaneous. In many cases Bu₄NI is used as a source of iodide anion that acts as a nucleophile toward carbon or other element centers. It is used to generate TMSI from TMSCl in situ in activated cyclopropane ring cleavage.⁸¹ Catalytic Bu₄NI activates iodosobenzene in the oxidation of tetrahydroisoquinoline alkaloids, conceivably via nucleophilic attack of iodide at iodoso groups.⁸² A 1:1 complex of Bu₄NI and NBS was suggested as a selective oxidant of alcohols.⁸³ Nucleophilic attack of iodide on the N–Cl group underlies the Bu₄NI catalysis in the aziridination of alkenes with chloramine-T and in unsaturated N-chloramine cyclization (eq 18).^{84,85}



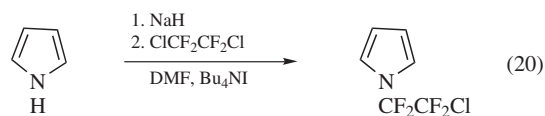
In the Zn(OTf)₂-catalyzed alkylation of indoles, the added Bu₄NI converted prenyl bromide to more active prenyl iodide, improving the β-regioselectivity.⁸⁶ Its effect in many other alkylation reactions with RX (X ≠ I) may be supposed to be of a similar nature.^{87–89} Alkylation and acylation of the pyrrole nitrogen (for protection purposes)^{90,91} requires the presence of Bu₄NI to proceed to completion.

Bu₄NI promotes glycosidation reactions, where it is superior to Bu₄NBr. It is used alone or in combination with 2,6-dialkylpyridinium salts or Ag₂CO₃ (eq 19).^{92–95}



Reaction of Bu₄NI with AgSCF₃ allowed generation of the unstable [SCF₃][−] anion for nucleophilic aromatic substitution reactions.⁹⁶

In the presence of Bu₄NI catalyst N-perfluoroalkylation of sodium azole salts was performed (eq 20).⁹⁷



Related Reagents. Tetra-*n*-butylammonium Bromide; Sodium Iodide; Lithium Iodide.

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