

Product Information

KitAlysis™ High Throughput Buchwald–Hartwig Amination Reaction Screening Kit

Catalog Number **KITALYSIS-CN**
Store at Room Temperature

TECHNICAL BULLETIN

Product Description

The KitAlysis™ High Throughput Buchwald–Hartwig Amination Reaction Screening Kit enables chemists to quickly and efficiently screen Buchwald–Hartwig C–N cross-coupling reaction conditions.



Quick identification of optimal conditions allows faster scale-up of the synthetic reaction. All required chemicals come pre-weighed for ease of use.

Each Buchwald–Hartwig Amination Reaction Screening Kit contains 4 individual screening sets. Each screening set has components to run 24 unique Buchwald–Hartwig amination reactions:

- 10 μmol of aryl halide (user supplied)
- 15–20 μmol of amine (primary or secondary, user supplied), use 20 μmol if substrate is not too precious
- 1 μmol of catalyst per vial (1:1 ratio of Pd:Ligand); 12 catalysts in each screening set
- 30 μmol of base (NaOt-Bu or Cs_2CO_3)
- 0.1 M solvent concentration (Dioxane or DMAc)

For each individual screening set, there are four possible protocols that can be run based upon the user's substrates:

- Two Solvents, Weak Base (Cs_2CO_3)
- Two Solvents, Strong Base (NaOt-Bu)
- One Solvent (Dioxane), Two Bases
- One Solvent (DMAc), Two Bases

Users can run four different protocols, four identical protocols, one of each, or any combination.

Protocol selection is based on the chemical nature of the substrates, see Figure 1.

Figure 1.
Protocol Selection

Nature of Substrates	Protocol	Attempt
Base sensitive	Two Solvents, Weak Base	First
Base tolerant	Two Solvents, Strong Base	First
Base tolerant	Dioxane, Two Bases	Second, based on substrate solubility
Base tolerant	DMAc, Two Bases	Second, based on substrate solubility
Not sure	Two Solvents, Strong Base	First
Not sure	Two Solvents, Weak Base	Second

Access www.sigmaaldrich.com/kitalysis for detailed step-by-step Protocols and Scale-up Guide. The Protocols include an introductory video, instructions for the Labware, and work-up procedures.

Downloadable spreadsheets for stock solution recipes for each screening set are linked from each Protocol. The spreadsheets provide:

- Calculations for substrate recipes based on the molecular weight of the substrates
- Quick directions for experienced users
- Print button allows you to take experimental recipes to the lab
- Contains all experimental information, which can easily be saved as a pdf file and appended to electronic laboratory notebooks

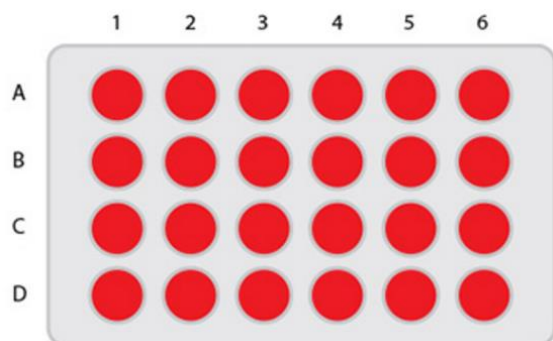
Screening Kit Components

- Four screening sets individually sealed in mylar bags. Each bag includes 24 glass vials loaded with stir bars and pre-weighed catalysts (1 μ mol), see Figures 2 and 3, topped with cap mat, 2 vials of pre-weighed ground and sieved cesium carbonate, and 2 empty reaction vials with lids
- Ampule boxes containing degassed, anhydrous liquids:
 - 10 \times 2 mL Dioxane
 - 10 \times 2 mL DMAc
 - 4 \times 0.5 mL NaOt-Bu Solution (2 M in THF)
- Biphenyl Internal Standard (30 mg)
- Four replacement films to ensure a tight, contaminant-free seal on the 24-well reaction block every time.
- Nine individually packed stir bars

Figure 2.
Catalysts

Description	Product No.	Vial
BrettPhos Pd G3	761605	A1, C1
CPhos Pd G3	763004	A2, C2
DavePhos Pd G3	804959	A3, C3
P(tBu ₃) Pd G2	756482	A4, C4
J009 Pd G3	747130	A5, C5
MorDalPhos Pd G3	792357	A6, C6
BINAP Pd G3	804967	B1, D1
RuPhos Pd G2	753246	B2, D2
SPhos Pd G2	753009	B3, D3
tBuXPhos Pd G3	762229	B4, D4
XantPhos Pd G3	763039	B5, D5
XPhos Pd G3	763381	B6, D6

Figure 3.
Positions of Catalysts



Reagents and Equipment Required but Not Provided

- 96-well plate for automated HPLC analysis (Catalog Number Z688711)
- 96-well plate cap mat for automated HPLC analysis (Catalog Number C1111)
- 10–100 μ L pipette (Catalog Number Z683809)
- 2–200 μ L pipette tip refill (Catalog Number Z640220)
- 100–1,000 μ L pipette (Catalog Number Z683825)
- 50–1,000 μ L pipette tip refill (Catalog Number Z640247)
- 2-inch needles for ease in ampule solvent extraction (Catalog Number Z192570)
- 1-mL syringe for accurate solvent volume extraction from ampule (Catalog Number Z230723)

Specially designed KitAlysis Labware allows set up in a hood without use of a glove box.

- KitAlysis Benchtop Inertion Box (Catalog Number Z742064)
- KitAlysis 24-well Reaction and Screwdriver (Catalog Number Z742107)

Instructions for the Labware can be accessed in the Articles section of the website product display page for the kit.

Precautions and Disclaimer

This product is for R&D use only, not for drug, household, or other uses. Please consult the Safety Data Sheet for information regarding hazards and safe handling practices.

Storage/Stability

Store the kit at room temperature.

KitAlysis is a trademark of Sigma-Aldrich Co. LLC.

ES,MAM 06/16-1