

## ■ ASSOCIATED CONTENT

### SI Supporting Information

The Supporting Information is available free of charge at <https://pubs.acs.org/doi/10.1021/acs.analchem.0c04726>.

Sensitivity of model Suzuki–Miyaura cross-coupling reaction, LC-MS analysis results, structures of probes and reagents, general workflow for Suzuki–Miyaura biotinylation, dose-dependent proteome labeling, gel-based ABPP, time- and temperature-dependent labeling, concentration-dependent labeling, Suzuki–Miyaura labeling, assessment of precatalyst compatibility with PBS and aryl halogens, and experimental procedures (PDF)

Cellular labeling, peptide records from the dual method labeling experiment with and without sp3 clean up, dual probe labeling experiment results, and BSA labeling experiment results (XLSX)

Competitive m-CSCP data, cysteine- and lysine-containing peptides labeled by two or more compounds with an MS1 ratio >3, and lysine-containing peptides labeled only by bifunctional compounds 15–18, 21, or 22 with an MS1 ratio >3 (XLSX)

SIM-XL results (XLSX)