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Rhodium(III)-Catalyzed C-H Benzylation of Indole's C3 Position with Aza-*o*-Quinone Methides

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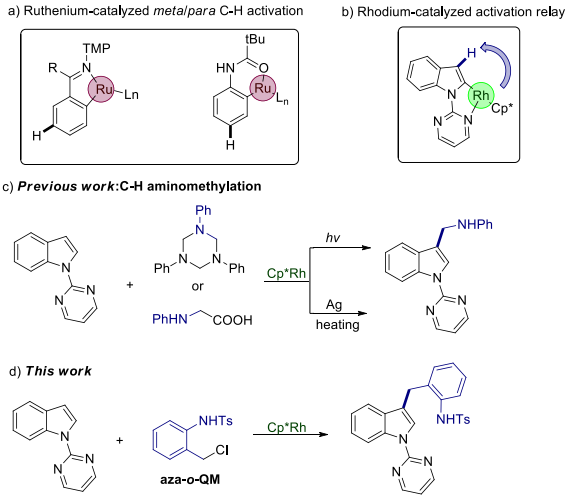
Abstract. In this paper, we report an example of indole's site selective C3 benzylation process through a rhodium(III)-catalyzed indole's C2 activation relay using aza-*o*-QM as a functionalization reagent, affording the desired products in good yields under mild conditions. The plausible reaction mechanism is proposed on the basis of control and deuterium labeling experiments and the further transformation has been also described.

Keywords: rhodium, C-H activation, quinone methides, benzylation, indoles;

Quinone methides (QMs) have been often utilized in organic chemistry as highly reactive intermediates for the facile synthesis of structurally diversified small molecules.[1] On the basis of their extraordinary reactivity and biological performance, the use of them in organic synthesis has recently attracted much attention. Moreover, they have been also extensively applied in total synthesis during the last decade.[2] In this aspect, Pettus and Water have reviewed QMs with regard to their preparations as well as the benefits and limitations in organic synthesis.[3] Their high reactivities are mainly due to their driving force for rapid rearomatization via Michael addition of nucleophiles, cycloadditions with a dienophile of 2π partners or 6π-electrocyclizations, especially in the cases of aza-*o*-QMs and *o*-QMs.[4]

Recently, C−H functionalization has been extensively explored as a versatile and highly efficient strategy for the synthesis of important skeletons implicated in organic compounds, natural products and pharmaceutical molecules.[5] Proximity-induced *ortho*-C–H functionalization is a widely-used strategy. Besides *ortho*-position, remote arene functionalizations are still challenges. Major recent progress are achieved by palladium,[6] iridium,[7] rhodium[8] and ruthenium[9] catalysis through steric control, template assistance, weak hydrogen bonding, transient mediators and other pathways. For example, ruthenium coordinated σ-activation is a typical strategy in accessing remote *meta*-selective functionalization. The key cyclometallated intermediates are shown in the Scheme 1a. Sterically crowded coupling partners or ligand sets suppress traditional *ortho*-functionalization pathways and promote complementary *meta*/*para*-functionalization. Meanwhile a free radical pathway can be commonly observed in ruthenium catalyzed *meta*/*para*-functionalization, indicating that the reaction partner producing radical is crucial to these reactions.



Scheme 1. Site-selective C–H Activation Reactions

The functionalization of indole has been extensively researched in the past years.[10] According to our previous work,[11,12] C–H bond of indole's C3 position can be activated via a rhodium complex chelated with *N*-tethered directing group at C2 position (Scheme 1b). Under this working framework, we have developed a series of aminomethylation methods with different reaction partners under photoredox catalysis[11] or upon heating[12] (Scheme 1c). In this context, we wish to report a novel Rh-catalyzed site-selective benzylation coupling of indole's C3 position with aza-*o*-QMs (Scheme 1d).

We initially used *N*-tosyl *ortho*-aminobenzyl chloride **2a** as a precursor of aza-*o*-QM to examine the reaction outcome. One molecule of hydrogen chloride can be eliminated from **2a** by adding a base to the reaction system, affording the reactive aza-*o*-

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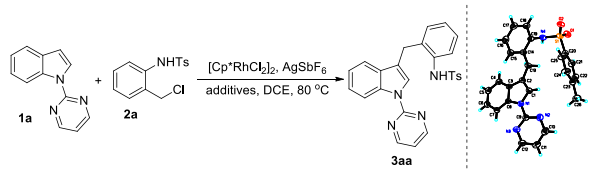
QM. The role of Ts protecting group is to stabilize the *in situ* generated aza-*o*-QM intermediate and also to enhance the acidity of hydrogen atom at the nitrogen center, rendering that the elimination can take place much easier using a regular base such as sodium carbonate.

Table 1. Optimization of the Reaction Conditions.



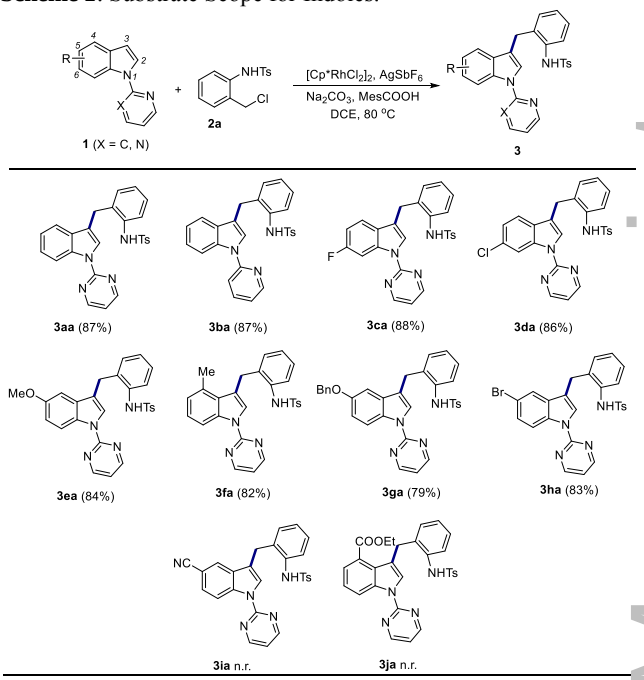
entry[a]	additive **1**	equiv	additive **2**	equiv	yield (%)[b]
1	Na_2CO_3	2.0	PivOH	2.0	67
2	Na_2CO_3	2.0	PhCOOH	2.0	55
3	Na_2CO_3	2.0	HOAc	2.0	61
4	Na_2CO_3	2.0	MesCOOH	2.0	87
5	Na_2CO_3	2.0	-	-	33
6	$NaHCO_3$	2.0	-	-	13
7	K_2CO_3	2.0	MesCOOH	2.0	n.r.
8	Cs_2CO_3	2.0	MesCOOH	2.0	n.r.
9	NaOAc	2.0	MesCOOH	2.0	43
10	NaOPiv	2.0	MesCOOH	2.0	58
11	Na_2CO_3	2.0	MesCOOH	1.5	77
12[c]	Na_2CO_3	2.0	MesCOOH	1.5	46
13[d]	Na_2CO_3	2.0	MesCOOH	1.5	n.r.

[a]Reaction conditions: **1a** (0.1 mmol), **2a** (0.12 mmol), $[Cp^*RhCl_2]_2$ (4 mol %) $AgSbF_6$ (20 mol %), acid (0.20 mmol), base (0.20 mmol), DCE (1.0 mL), the reaction was carried out at 80 °C for 12 h, ambient atmosphere; [b]Isolated yield; [c] carried out in THF; [d] carried out in MeOH, TFE and HFIP.

The reaction conditions were optimized by employing easily prepared indole **1a** as a model substrate for the C−H benzylation with precursor **2a** using $[Cp^*RhCl_2]_2$ and $AgSbF_6$ as the catalyst combination in 1,2-dichloroethane (DCE) at 80 °C and the results are summarized in Table 1. We found that the desired product **3aa** was obtained in 67% yield in the presence of Na_2CO_3 (2.0 equiv) and pivalic acid (PivOH) (2.0 equiv) (entry 1). The structure of **3aa** was confirmed by the X-ray crystallographic analysis and its ORTREP drawing is shown in Table 1.

We then screened several different acids and bases involved in the reaction and examined their equivalents to achieve the higher yield of **3aa**. After examining several acids, we found that using benzoic acid and acetic acid in the reaction afforded **3aa** in 55% and 61% yields, respectively, which were similar as that of using pivalic acid in the reaction (entries 2-3). However, the use of 2,4,6-trimethylbenzoic acid could give **3aa** in the yield of 87% (entry 4). Noteworthy, the yield of **3aa** decreased dramatically to 33% if without acidic additive, suggesting that an acidic additive must be required to enhance the C-H activation efficiency in the reaction (entry 5). Next, we also examined different bases in the reaction, which may relate to the production of aza-*o*-QM. We found that none of **3aa** could be obtained if using stronger inorganic bases such as potassium carbonate (K_2CO_3) and cesium carbonate in the reaction (Cs_2CO_3) (entries 7-8). These stronger bases probably lead to the decomposition of QM. The utilization of $NaHCO_3$, sodium acetate or sodium pivalate, that had the weaker basicity than that of Na_2CO_3, as a base in the reaction also reduced the yield of **3aa** to 13%, 43% and 58%, respectively (entries 6, 9-10). It should be emphasized here that MesCOOH was completely dissolved in DCE, whereas Na_2CO_3 was only partially dissolved in it, leading to the reaction solution to be acidic. Lowering the employed amount of 2,4,6-trimethylbenzoic acid to 1.5 equiv provided **3aa** in 77% yield (entry 11), indicating that this reaction was facilitated under acidic conditions. The reaction cannot happen in the alcohol solvent and yield decreased dramatically in THF. (entries 12-13, for more information, see Table S1 in the Supporting Information)

Scheme 2. Substrate Scope for Indoles.[a,b]



[a]Reaction conditions: **1** (0.1 mmol), **2a** (0.12 mmol), $[Cp^*RhCl_2]_2$ (4 mol %) $AgSbF_6$ (20 mol %), Na_2CO_3 (0.20 mmol), MesCOOH (0.20 mmol), DCE (1.0 mL), the reaction was carried out at 80 °C for 12 h, ambient atmosphere; [b]Isolated yield.

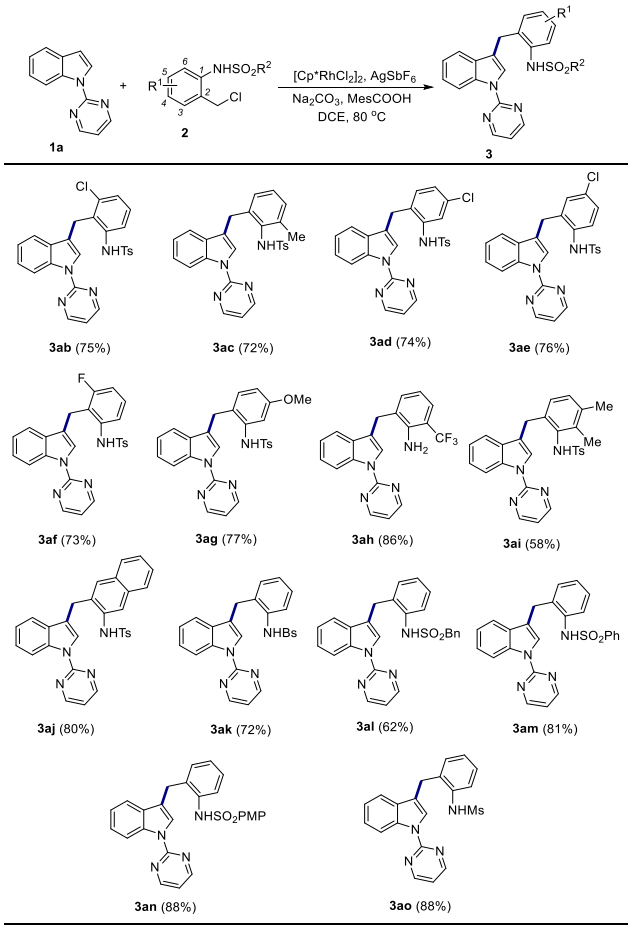
After establishing the optimal reaction conditions, we next investigated the scope of indole substrates bearing various substituents. The results are shown in Scheme 2. When the pyrimidine group is replaced with a pyridine, the reaction also proceeded smoothly, giving the desired product **3ba** in 87% yield. Substrates **1c** and **1d** having halogen substituents at the C6 position of indole moiety underwent the reaction smoothly, affording the corresponding products **3ca** and **3da** in 88% and 86% yields, respectively. Introducing OMe group (substrate **1e**), OBn group (substrate **1g**) and bromine atom (substrate **1h**) into the C5 position of indole moiety, the reactions also went through very well, delivering the desired products **3ea**, **3ga** and **3ha** in 82% to 84% yields. For a methyl substituent (substrate **1f**) at the C4 position of indole moiety, the reaction was also tolerated, furnishing the product **3fa** in 82% yield. Other unsaturated functional group like CN and COOEt are not compatible in this reaction (**3ia** and **3ja**).

Next, we examined the generality of various substituents on the benzene ring of aza-*o*-QMs, and the results are depicted in Scheme 3. With the introduction of chlorine atom at the 3, 4 and 5 positions of QM precursor (substrates **2b**, **2d** and **2e**), the reactions proceeded efficiently, giving the desired products **3ab**, **3ad** and **3ae** in 74-76% yields. Substrate **2c** containing a methyl group at the 6 position of QM precursor, substrate **2f** bearing a

fluorine atom at the 3 position of QM precursor, and substrate **2g** having a methoxy group at the 5 position of QM precursor were all well tolerated, affording the corresponding products **3ac**, **3af** and **3ag** in good yields ranging from 72% to 77%. In the case of substrate **2h** bearing a trifluoromethyl group at the 6 position of QM precursor, the detosylated product **3ah** was obtained in 86% yield presumably due to that the electron-deficient benzene ring caused the instability of the sulfonyl group in QM precursor under the standard conditions. Introducing two methyl groups on the benzene ring of QM precursor or the QM precursor containing a naphthyl group were also compatible for this reaction, giving the desired products **3ai** and **3aj** in 58% and 80% yields, respectively.

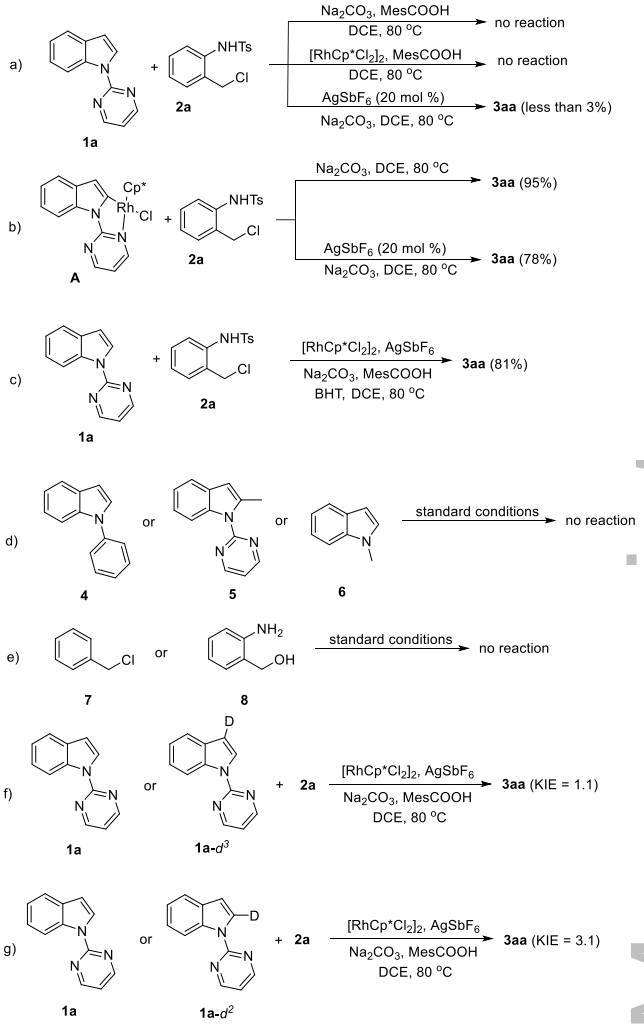
In addition, we also investigated the effect of the sulfonyl group on QM precursors. Replacing Ts with *para*-bromobenzenesulfonyl group (substrate **2k**), benzylsulfonyl group (substrate **2l**), benzenesulfonyl group (substrate **2m**), *para*-methoxybenzenesulfonyl group (substrate **2n**) and methanesulfonyl group (substrate **2o**), all the reactions could furnish the corresponding products **3ak**-**3ao** in good yields ranging from 62% to 88%, indicating a broad substrate scope.

Scheme 3. Substrate Scope for aza-*o*-QMs.[a,b]



[a]Reaction conditions: **1a** (0.1 mmol), **2** (0.12 mmol), $[Cp^*RhCl_2]_2$ (4 mol %) $AgSbF_6$ (20 mol %), Na_2CO_3 (0.20 mmol), MesCOOH (0.20 mmol), DCE (1.0 mL), the reaction was carried out at 80 °C for 12 h, ambient atmosphere; [b]Isolated yield.

Scheme 4. Control Experiments

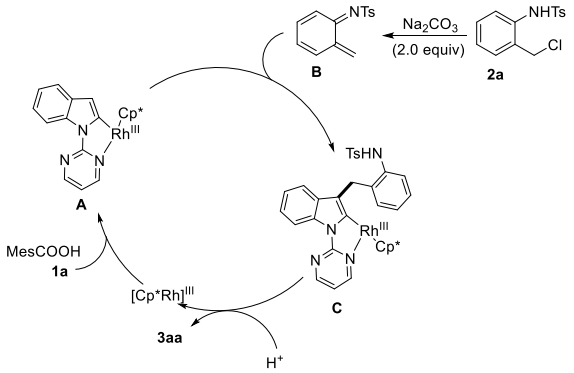


Several control experiments were performed to clarify the necessity of the catalysts used in the reaction (Scheme 4). No reaction occurred in the absence of $[Cp^*RhCl_2]_2$ and $AgSbF_6$ and trace of **3aa** was formed if only using $AgSbF_6$ as the catalyst, suggesting that the catalyst combination of $[Cp^*RhCl_2]_2$ and $AgSbF_6$ is essential for the reaction (Scheme 4a). According to the previous literature,[13] we synthesized indole's *ortho*-C2–H metallated complex **A** to validate the role of $AgSbF_6$ in the reaction with aza-*o*-QM intermediate (Scheme 4b). Upon treating Rh complex **A** with **2a** afforded **3aa** in 95% yield in the presence of Na_2CO_3, and the extra addition of $AgSbF_6$ (20 mol %) did not improve the yield of **3aa**, suggesting that $AgSbF_6$ is not necessary for the reaction of complex **A** with aza-*o*-QM intermediate. $AgSbF_6$ as Lewis acid probably obstructs the role of Na_2CO_3; and it may grab chloride of rhodium center leading to the decomposition of complex **A**. The radical inhibitor BHT did not affect the yield of **3aa**, which may exclude the radical process (Scheme 4c). To verify the activation relay process, we used substrate **4**, in which no directing group was included, and substrate **5**, in which a methyl group was introduced at indole's C2 position, and *N*-Me indole **6** under the standard conditions and found that none of them could give any desired product (Scheme 4d). Benzyl chloride **7** and (2-aminophenyl)-methanol **8** are not reactive as well (Scheme 4e). These results indicated that the cationic rhodium-catalyzed activation relay is a crucial step for the reaction and the Lewis acid catalysis can be excluded from

this cascade transformation. Overall, these control experiments demonstrate that the *ortho*-C2–H metalation plays an important role in accessing C3–H functionalization.

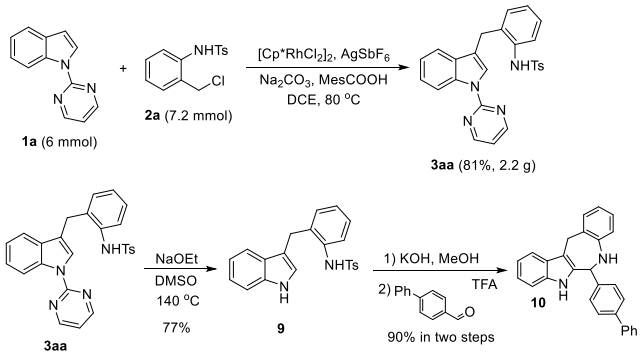
The determination of KIE for this reaction has been performed with **1a-*d*^{3}** and **1a-*d*^{2}** as substrates under the standard conditions (Schemes 4f and 4g). The kinetic isotope effect observed in parallel reactions using **1a** and **1a-*d*^{3}** as substrates was 1.1, suggesting that benzylation at the C3 position is not a rate-determining step. In contrast, a larger kinetic isotope effect (KIE = 3.1) was observed in the parallel reactions using **1a** and **1a-*d*^{2}** as substrates, indicating that the C-H bond activation at indole's C2 position might be the rate-determining step.

Scheme 5. Proposed Reaction Mechanism



According to the previous literature and our examinations mentioned above, a plausible mechanistic paradigm was proposed in Scheme 5. The nitrogen atom coordinates to a cationic $Cp^*Rh(III)$ species to form a rhodacyclic complex **A** via the cyclometalation at the proximal position,[13] which probably could be assisted and accelerated by MesCOOH additive. Meanwhile, aza *o*-QM intermediate **B** is *in situ* produced from *N*-tosyl *ortho*-aminobenzyl chloride **2a** along with the elimination of hydrogen chloride in the presence of Na_2CO_3. The reaction of rhodium complex **A** with **B** affords intermediate **C** through a Michael addition reaction, which undergoes a proton-demetalation by acid to give the desired product **3aa** and regenerates the Rh(III) catalyst. In this reaction, Ag salt reacts with $[Cp^*RhCl_2]_2$ to yield active cationic rhodium catalytic species. Without silver assistance, $[Cp^*RhCl_2]_2$ is hard to activate substrate C-H bond efficiently. It dose not play a role in the catalytic circle.

Scheme 6. Synthetic Application and Scale-up Reaction



Furthermore, this transformation could be conducted at 6.0 mmol scale, affording **3aa** in 81% yield (Scheme 6). The pyrimidine group in **3aa** could be easily removed by treating with NaOEt in DMSO at 140 °C to give the corresponding product **9** in 77% yield, which could be further transformed to the cyclized product **10** upon removing Ts group with KOH in methanol and treating with biphenylcarboxaldehyde in TFA through a Pictet-Spengler reaction (Scheme 6).

In conclusion, we have developed a novel Rh-catalyzed benzylation protocol by adopting aza-*o*-QMs as a reagent through C-H bond activation. The site selective benzylation for C-H bond at indole's C3 position can be deemed as another example in Rh-catalyzed activation relay domain. The obtained products have diversified functional groups and exhibit good compatibilities in further transformations. Efforts to extend the realm of Rh-catalyzed activation relay are undergoing in our lab.

Experimental Section

General Procedure for Synthesis of 3

To a flame dried reaction flask was added **1a** (0.1 mmol), **2a** (0.1 mmol), $[RhCp^*Cl_2]_2$ (0.004 mmol), $AgSbF_6$ (0.02 mmol), Na_2CO_3 (0.2 mmol), MesCOOH (0.2 mmol). Then DCE (1.0 mL) was added to the reaction mixture using a syringe. The reaction was carried out at 80 °C. After the reaction was complete, the solution was concentrated under reduced pressure and the residue was purified with a silica gel column chromatography to afford the product.

Supporting Information Available

Detailed descriptions of experimental procedures and their spectroscopic data as well as the crystal structures are presented in the Supporting Information. CCDC 1936773 (**3aa**) contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Center via www.ccdc.cam.ac.uk/data_request/cif.

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UPDATE

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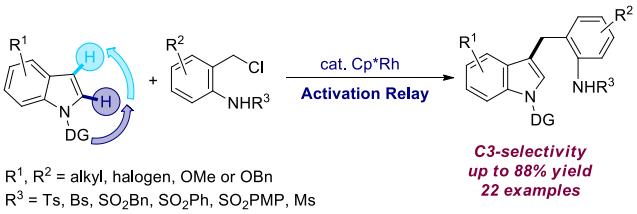

R^1, R^2 = alkyl, halogen, OMe or OBn
R^3 = Ts, Bs, SO_2Bn, SO_2Ph, SO_2PMP, Ms

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