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# Cyclization of *ortho*-alkynylphenylphosphine *P*-ylides; dependence on ylide nucleophilicity

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ABSTRACT

P=$CH_2$ ylides derived from *ortho*-alkynylphenylphosphines readily cyclize to give phosphindole derivatives. With a stabilized ylide such as P=CHC(O)Ph, the outcome of the reaction is completely different. The reaction proceeds at high temperature and produces phosphanaphthalenes.

## 1. Introduction

The nucleophilic phosphonium ylides (P-ylides) are one of the most widely used classes of compound in modern chemistry. Besides the well known Wittig reaction [1,2], P-ylides have been used as nucleophiles in many coupling reactions with electron-deficient alkenes [3–7] or alkynes [8,9], acyl chlorides [10–12], propargylic acetate [13], allyl carbonates [14], imines or esters [15–17] and $CO_2$ [18] et al. [19–27], as ligands in coordination chemistry [28–32] or as catalysts [33–35]. In contrast to those well-established reactions of P-ylides, to our knowledge, the reaction of P-ylides with unactivated alkynes has never been explored. In a series of papers, we have recently described several synthetic applications of *ortho*-alkynylphenylphosphine derivatives [36–39]. Hereafter, we wish to report on our observations concerning the ylides derived from these phosphines.

## 2. Results and discussion

The attempted synthesis of the starting ylides was performed as shown in Scheme 1.

We were struck by the fact that the expected ylides **2** are highly unstable and rapidly cyclize at room temperature to give the cyclic ylides **3** in high yields. The products were characterized as the phosphonium salts **4**. The structure of **4a** is shown in figure (1).

As seen, the cyclization produces exclusively the compound resulting from the nucleophilic attack of the ylidic carbon at the α position of the triple bond with the $R^1$ substituent in the *endo* position. Since the resulting ylides **3** are somewhat strained and not stabilized by electronic delocalization, we were a little bit surprised by the easiness of this cyclization. We decided to have a closer look at the geometry and electronic structure of the precursor ylide **2a**. The computations were carried out by DFT at the B3LYP/6-31G(d) level [40]. The HOMO and the LUMO of **2a** are shown in figure (2). The HOMO is massively localized at the ylidic carbon as expected and the LUMO is significantly located on both $C_\alpha$ and $C_\beta$. But the DFT calculations indicated that $C_{ylidic}$-$C_\alpha$ is shorter than $C_{ylidic}$-$C_\beta$ (3.87 Å). The regioselectivity of the cyclization for $C_\alpha$ has a purely spatial proximity origin.

It is interesting to note here that a somewhat similar cyclization has been described in the literature with the C≡C triple bond replaced by an isocyanide N≡C triple bond [41]. Since the cyclization results from the nucleophilic attack of the ylidic carbon at $C_\alpha$, it was interesting to check what would happen with a stabilized ylide of reduced nucleophilicity. Using a benzoyl substituted ylide, the

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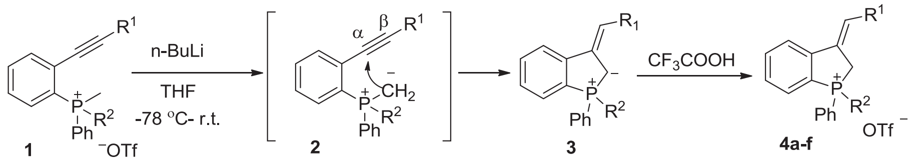


| Entry | $R^1$ | $R^2$ | Product | Isolated Yield% |
|---|---|---|---|---|
| 1 | Ph | Ph | **4a** | 84 |
| 2 | 4-Methoxyphenyl | Ph | **4b** | 82 |
| 3 | 1-Naphthyl | Ph | **4c** | 86 |
| 4 | benzo[*b*]thiophen-2-yl | Ph | **4d** | 80 |
| 5 | *n*-Bu | Ph | **4e** | 73 |
| 6 | Ph | $^t$Bu | **4f** | 86 |

**Scheme 1.** Synthesis and cyclization of ylides.

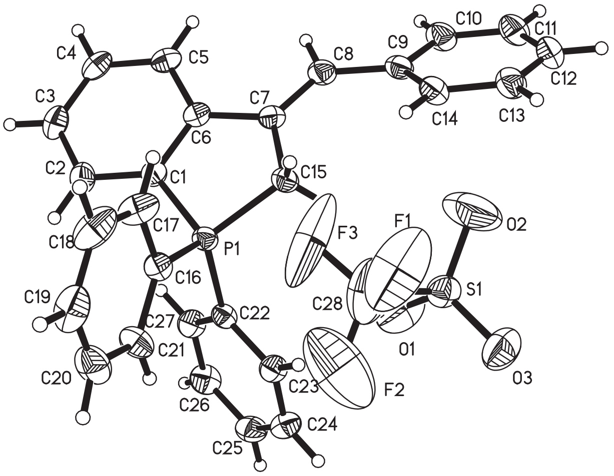


**Fig. 1.** X-ray crystal structure of product **4a**.

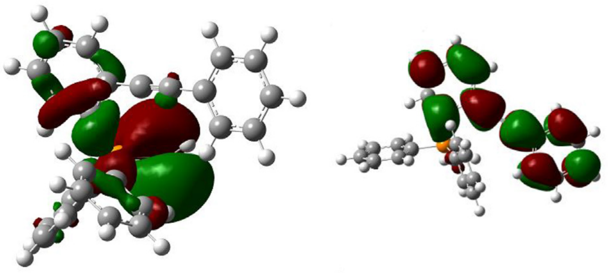
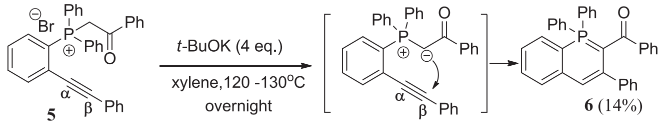


**Fig. 2.** Frontier orbitals of **2a**: HOMO left, LUMO right.

outcome of the reaction is completely different. No reaction was observed at room temperature. At higher temperature, a phosphanaphthalene **6** was produced (Scheme 2). To the best of our knowledge, there is only limited methods have been developed for the synthesis of phosphanaphthalenes [42–51].

The product **6** was identified by X-ray crystal structure analysis (Fig. 3).

In our case, no reaction is observed at room temperature because the nucleophilicity of the ylidic carbon is too low. At high temperature, we guess that the reaction is not anymore under orbital control. A NBO analysis of the ylide from **5** shows that the C≡C bond is somewhat polarized: $C_\alpha = -0.012$, $C_\beta = +0.035$. The ylidic carbon tends to react at $C_\beta$ which is positively charged. The high temperature and the low yield reflect the difficulty of the reaction. In order to confirm this electronic effect, an electron donating methoxy group was introduced to reduce the electrophilicity of the alkyne. No cyclization was observed with the corresponding ylide, even at elevated temperature. But the reactivity of the C≡C bond towards the stabilized ylide could be boosted by addition of 1 equivalent of AuCl(THT) as Lewis acid, and the cyclization proceeded smoothly to provide the desired product **8** with shorter reaction time (3 h) and higher yield (46%, compared with **6**) (Scheme 3).



**Scheme 2.** Synthesis of a phosphanaphthalene.

## 3. Conclusions

Anyhow, the complete shift from one reaction pathway to another upon decreasing the nucleophilicity of the ylide is certainly the most striking result of this study. The calculation indicated that

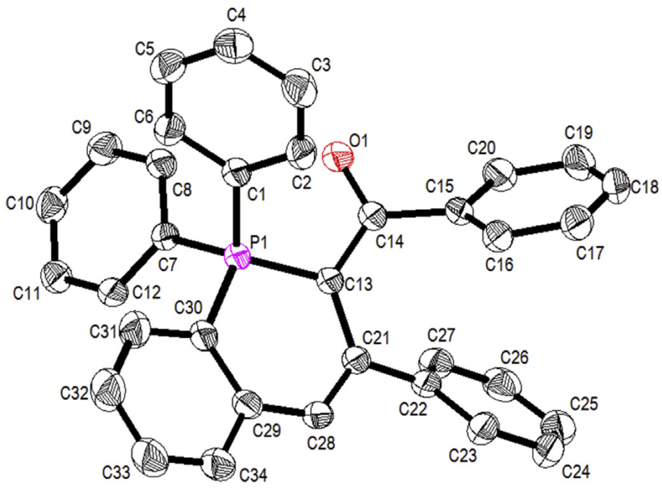
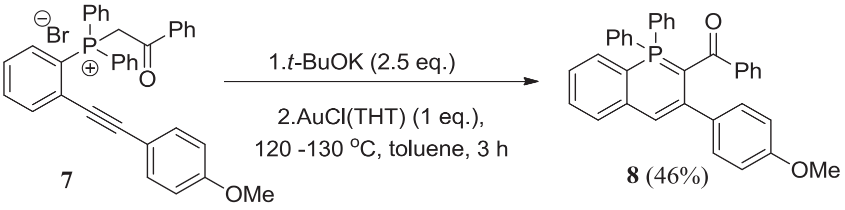


**Fig. 3.** X-ray crystal structure of product **6**.



**Scheme 3.** Lewis acid promoted cyclization.

spatial proximity plays an important role for the highly nucleophilic ylide, while the selectivity of the stabilized ylide was mainly controlled by the electron deficiency of the alkyne. To the best of our knowledge, this observation is unprecedented in ylide chemistry.

## 4. Experimental

All reactions were routinely performed under nitrogen by using standard Schlenk techniques and dry deoxygenated solvents. Dry THF was obtained by distillation from Na/benzophenone. Dry toluene was obtained by distillation from $P_4O_{10}$. NMR spectra were recorded with a Bruker 300 MHz spectrometer operating at 300.13 MHz for$^1$H, 75.468 MHz for $^{13}$C, and 121.495 MHz for $^{31}$P NMR spectroscopy. Chemical shifts are given in ppm relative to internal TMS ($^1$H and $^{13}$C). All coupling constants (J values) are reported in Hertz [Hz]. HRMS data were recorded with an Agilent 1290-6540 Q-Tof spectrometer by electrospray ionization (ESI).

### 4.1. Synthesis of **1a**

Methyl trifluoromethanesulfonate(272 µL, 2.4 mmol) was added to a stirred solution of diphenyl(2-(phenylethynyl)phenyl)phosphane(725 mg, 2 mmol) in dichloromethane (30 mL) at room temperature. After stirring of the mixture for 2 h, the solvent was evaporated to give the product. White solid. $^{31}$P NMR (300 MHz, $CDCl_3$): δ 22.45. $^1$H NMR (300 MHz, $CDCl_3$): δ 3.03 (d, $J_{H-P} = 27$ Hz, 3H, $PCH_3$), 6.05 (d, $J_{H-H} = 1.5$ Hz, 2H), 6.93–7.35 (m, 4H, Ph), 7.55–7.61 (m, 1H, Ph), 7.65–7.86 (m, 12H, Ph). $^{13}$C NMR (300 MHz, $CDCl_3$): δ 8.97 (d, $J_{C-P} = 58.3$ Hz, $PCH_3$), δ 86.24 (d, $J_{C-P} = 6.6$ Hz, $C_{alkyne}$), 100.92 (s, $C_{alkyne}$), 118.45(d, $J_{C-P} = 89.1$ Hz, $PC_{ar}$), 119.62 (d, $J_{C-P} = 85.3$ Hz, $PC_{ar}$), 120.37 (s, $CH_{ar}$),127.48 (d, $J_{C-P} = 7.5$ Hz, $CH_{ar}$), 128.60 (s, $C_{ar}$), 129.78 (d, $J_{C-P} = 12.7$ Hz, $CH_{ar}$), 129. 92 (s, $CH_{ar}$), 130.52 (d, $J_{C-P} = 12.9$ Hz, $CH_{ar}$), 131.16 (s, $CH_{ar}$), 132.98 (d, $J_{C-P} = 10.6$ Hz, $CH_{ar}$), 135.11 (d, $J_{C-P} = 3$ Hz, $CH_{ar}$), 135.19 (d, $J_{C-P} = 2.6$ Hz, $CH_{ar}$), 135.28(d, $J_{C-P} = 2.4$ Hz, $CH_{ar}$), 135.42(d, $J_{C-P} = 11$ Hz, $CH_{ar}$). HRMS (FAB+) calcd for $C_{27}H_{22}P$, 377.1453; found, 377.1456.

### 4.2. Synthesis of **4a**

Under nitrogen, *n*-Buti (0.65 ml, 1.6 M, 1.05 mmol)was added to THF (10 mL) solution of 1 (1.0 mmol) at −78 °C. The mixture was warmed to room temperature and stirred for 1 h, and then Trifluoroacetic acid (171 mg, 1.5 mmol) was added. After evaporation of the solvent, the crude mixture was recrystallized with dichloromethane and *n*-hexane to afford the white solid **4a**, yield 84%. $^{31}$P NMR (121 MHz, $CDCl_3$): δ 34.23. $^1$H NMR (300 MHz, $CDCl_3$): δ 4.47 (dd, $J_{H-P} = 12$ Hz, $J_{H-H} = 1.8$ Hz, 2H, $PCH_2$), 7.28–7.35 (m, 1H, Ph), 7.42–7.52 (m, 3H, Ph), 7.61–7.93 (m, 15H, Ph), 8.06 (dd, $J_{H-P} = 8.1$ Hz, $J_{H-H} = 3.0$ Hz, 1H, Ph). $^{13}$C NMR (75 MHz, $CDCl_3$): δ 29.96 (d, $J_{C-P} = 57.9$ Hz, $PCH_2$), 118.19 (d, $J_{C-P} = 85.5$ Hz, $PC_{ar}$), 119.74 (d, $J_{C-P} = 86.9$ Hz, PC), 123.43 (d, $J_{C-P} = 11.4$ Hz, CH), 128.63 (d, $J_{C-P} = 4.5$ Hz, $C_{ar}$), 129.12 (s, $CH_{ar}$), 129.19 (s, $CH_{ar}$), 129.67 (s, $CH_{ar}$), 130.48 (d, $J_{C-P} = 13.2$ Hz, $CH_{ar}$), 130.72 (d, $J_{C-P} = 10.7$ Hz, $CH_{ar}$), 130.89 (d, $J_{C-P} = 11$ Hz, $CH_{ar}$), 131.09 (d, $J_{C-P} = 8.8$ Hz, $CH_{ar}$), 133.22(d, $J_{C-P} = 11$ Hz, $CH_{ar}$), 134.59 (s, $C_{ar}$), 135.32 (d, $J_{C-P} = 3.1$ Hz, $CH_{ar}$), 136.19 (d, $J_{C-P} = 2.3$ Hz, $CH_{ar}$), 149.22 (d, $J_{C-P} = 22.3$ Hz, $C_{ar}$); HRMS (FAB+) calcd for $C_{27}H_{22}P$, 377.1453; found, 377.1459. **4b,f** were prepared similarly (see supplementary data).

### 4.3. Synthesis of **5**

2-Bromo-1-phenylethan-1-one (1.09 g, 5.5 mmol) was added to a THF (20 mL) solution of diphenyl(2-(phenylethynyl)phenyl) phosphane (1.81 g, 5 mmol) at room temperature. The reaction mixture was stirred at 60 °C for overnight. After evaporation of the solvent, the crude product was washed by diethyl ether and **5** was btained as a white solid, yield 80%. $^{31}$P NMR (121 MHz, $CDCl_3$): δ 22.88. $^1$H NMR (300 MHz, $CDCl_3$): δ 6.46 (d, $J = 12.0$ Hz, 2H, $CH_2$), 6.72–6.74 (m, 2H, Ph), 7.19–7.31 (m, 5H, Ph), 7.51–7.74 (m, 11H, Ph), 7.98–8.12 (m, 6H, Ph). $^{13}$C NMR ($CDCl_3$): δ 39.68(d, $J_{C-P} = 61.0$ Hz, $CH_2$), 86.33(d, $J_{C-P} = 6.6$ Hz, C), 100.10 (s, C), 118.42(d, $J_{C-P} = 89.7$ Hz, C), 119.66(d, $J_{C-P} = 90.7$ Hz, C), 120.05(s,C), 126.50(d, $J_{C-P} = 6.8$ Hz, C), 128.47(s, CH), 128.62(s, CH), 129.23(s, CH), 129.56(d, $J_{C-P} = 13.0$ Hz, CH), 129.73(s, CH), 129.98(d, $J_{C-P} = 13.1$ Hz, CH), 130.94(s, CH), 134.09(d, $J_{C-P} = 10.8$ Hz, CH), 134.33(s, CH), 134.59(s, CH), 134.62(s, CH), 134.76(d, $J_{C-P} = 9.6$ Hz, CH), 135.09(d, $J_{C-P} = 5.63$ Hz, C), 135.83(d, $J_{C-P} = 10.5$ Hz, CH), 192.04(d, $J_{C-P} = 6.3$ Hz, C=O). HRMS Calcd. For $C_{34}H_{26}OP^+$ $[M - Br^-]^+$, 481.1716. Found: 481.1720.

### 4.4. Synthesis of **6**

Under nitrogen, potassium tert-butoxide (448 mg, 4.0 mmol) and 3 activated molecular sieves were added to a xylene (3 mL) solution of **5** (561 mg, 1 mmol, 1eq) at room temperature. The white solid was dissolved to give a dark red viscous solution. The reaction mixture was stirred at 120–130 °C for overnight. After evaporation of the solvent, the crude product was purified by chromatography on neutral alumina (PE: EA = 100:1-20:1) and **6** was obtained as a yellow solid, yield 14%. $^{31}$P NMR (121 MHz, $CDCl_3$): δ 7.48. $^1$H NMR (300 MHz, $CDCl_3$): δ 5.96 (d, $J = 3.6$ Hz, 1H), 6.86–7.00 (m, 6H), 7.08–7.13 (m, 1H), 7.19–7.25 (m, 3H), 7.31–7.37 (m, 3H), 7.43–7.53 (m, 7H), 7.78–7.92 (m, 4H).$^{13}$C NMR (75 MHz, $CDCl_3$): δ 70.80(d, $J_{C-P} = 102.8$ Hz, P=C), 110.94(d, $J_{C-P} = 10.5$ Hz, CH), 111.16(d, $J_{C-P} = 87.0$ Hz, C), 124.25(d, $J_{C-P} = 11.3$ Hz, CH), 126.49(s, CH), 126.91(s, CH), 127.15(d, $J_{C-P} = 8.3$ Hz, CH), 127.40(s, CH), 127.79(d, $J_{C-P} = 91.5$ Hz, C),128.65(d, $J_{C-P} = 12.75$ Hz, CH), 128.87(s, CH),129.01(s, CH), 129.14(s, CH), 131.58(s, CH), 131.60(d, $J_{C-P} = 3.0$ Hz, CH), 132.40(d, $J_{C-P} = 7.5$ Hz, CH), 133.05(d, $J_{C-P} = 11.3$ Hz, CH), 141.59(d, $J_{C-P} = 5.3$ Hz, C), 142.43(d, $J_{C-P} = 9.8$ Hz, C), 144.05(d, $J_{C-P} = 8.3$ Hz, C), 144.11(d, $J_{C-P} = 11.3$ Hz, C), 189.66(d, $J_{C-P} = 6.0$ Hz, C=O). HRMS Calcd. For $C_{34}H_{25}OP$ $[M + H]^+$, 481.1716. Found: 481.1720.

### 4.5. Synthesis of **7**

2-Bromo-1-phenylethan-1-one (1.09 g, 5.5 mmol) was added to a THF (20 mL) solution of (2-((4-methoxyphenyl)ethynyl)phenyl)diphenylphosphane (1.96, 5 mmol) at room temperature. The reaction mixture was stirred at 60 °C for overnight. After evaporation of the solvent, the crude product was washed by diethyl ether and 7 was obtained as a yellow solid, yield 72%. $^{31}P$ NMR (121 MHz, $CDCl_3$): δ 23.01. $^{1}H$ NMR (300 MHz, $CDCl_3$): δ 3.72 (s, 3H, $OCH_3$), 6.36 (d, $J = 12.0$ Hz, 2H, $CH_2$), 6.60–6.67 (m, 4H, Ph), 7.25–7.30 (m, 2H, Ph), 7.42–7.69 (m, 11H, Ph), 7.91–7.98 (m, 4H, Ph), 8.05–8.08 (m, 2H, Ph). $^{13}C$ NMR ($CDCl_3$): δ 39.82(d, $J_{C\text{-}P} = 61.5$ Hz, $CH_2$), 55.43(s, $OCH_3$), 85.55(d, $J_{C\text{-}P} = 6.8$ Hz, C), 100.95 (s, C), 112.11(s, C), 114.30(s, CH), 118.70(d, $J_{C\text{-}P} = 89.3$ Hz, C), 119.76(d, $J_{C\text{-}P} = 90.0$ Hz, C), 127.28(d, $J_{C\text{-}P} = 7.5$ Hz, C), 128.74(s,CH), 129.14(d, $J_{C\text{-}P} = 12.9$ Hz, CH), 129.48(s, CH), 130.07(d, $J_{C\text{-}P} = 13.2$ Hz, CH), 132.80(s, CH), 134.22(s, CH), 134.37(s, CH), 134.41(s, CH), 134.54(d, $J_{C\text{-}P} = 12.5$ Hz, CH), 134.58(s, CH), 135.23(d, $J_{C\text{-}P} = 6.0$ Hz, C), 135.87(d, $J_{C\text{-}P} = 10.5$ Hz, CH), 160.74(s, C), 192.29(d, $J_{C\text{-}P} = 6.8$ Hz, C=O). HRMS Calcd. For $C_{35}H_{28}O_2P^+$ $[M - Br^-]^+$, 511.1821. Found: 511.1822.

### 4.6. *Synthesis of* ***8***

Under nitrogen, potassium tert-butoxide (56 mg, 0.5 mmol, 2.5eq) were added to a xylene (3 mL) solution of **7** (119 mg, 0.2 mmol, 1eq) at room temperature. The yellow solid was dissolved to give a red viscous solution. AuCl(THT) (64 mg, 0.2 mmol, 1eq) was added to the solution. The reaction mixture was stirred at 120–130 °C for 3 h. After evaporation of the solvent, the crude product was purified by chromatography on neutral alumina (petroleum ether: ethyl acetate = 100:1-20:1) and **8** was obtained as a yellow solid, yield 46%.$^{31}P$ NMR (121 MHz, $CDCl_3$): δ 7.99. $^{1}H$ NMR (300 MHz, $CDCl_3$): δ 3.66 (s, 3H, $OCH_3$), 5.94(d, $J_{H\text{-}H} = 3.3$ Hz, 1H), 6.50–6.53 (m, 2H), 6.92–7.02 (m, 3H), 7.08–7.23 (m, 4H), 7.32–7.38 (m, 3H), 7.44–7.54 (m, 7H), 7.82–7.90 (m, 4H).$^{13}C$ NMR (75 MHz, $CDCl_3$): δ 55.23(s, $OCH_3$), 72.08(d, $J_{C\text{-}P} = 103.5$ Hz, P=C), 110.03(d, $J_{C\text{-}P} = 9.6$ Hz, CH), 110.97(d, $J_{C\text{-}P} = 87.3$ Hz, C), 112.93(s, CH), 123.95(d, $J_{C\text{-}P} = 11.8$ Hz, CH), 126.88(s, CH), 126.96(d, $J_{C\text{-}P} = 10.5$ Hz, CH), 127.87(d, $J_{C\text{-}P} = 91.6$ Hz, C), 128.61(d, $J_{C\text{-}P} = 12.8$ Hz, 2CH), 128.82(s, CH), 129.09(s, CH), 130.11(s, CH), 131.55(d, $J_{C\text{-}P} = 2.8$ Hz, CH), 132.37(d, $J_{C\text{-}P} = 6.8$ Hz, CH), 133.03(d, $J_{C\text{-}P} = 10.7$ Hz, CH), 136.83(d, $J_{C\text{-}P} = 11.3$ Hz, C), 141.77(d, $J_{C\text{-}P} = 5.3$ Hz, C),142.45(d, $J_{C\text{-}P} = 9.8$ Hz, C), 143.65(d, $J_{C\text{-}P} = 9.0$ Hz, C), 158.31(s, C), 189.70(d, $J_{C\text{-}P} = 6.0$ Hz, C=O). HRMS Calcd. For $C_{35}H_{28}O_2P$ $[M + H]^+$, 511.1821. Found: 511.1820.

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## Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.jorganchem.2018.09.023.

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