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# SYNTHESIS OF α-BROMOMETHYL KETONES IN CuBr - bpy SYSTEM

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**Abstract** – Herein we reported a convenient, regiospecific, efficient and fairly general method for the preparation of α-bromomethyl ketones from α,α,α-tribromomethyl carbinols with 2.2 equivalents of copper(I) bromide and 2,2'-bipyridine in refluxing 1,2-dichloroethane.

Heterocyclic compounds are recognized as important components of organic chemistry and important building blocks because of their universality in numerous natural products, agrochemical and pharmaceutical industries and material science.[1,2] Among all these heterocyclic compounds, several heterocyclic ketones are used as potential histone deacetylase inhibitors.[3] On the other hand, halogenated organic compounds are widely used as starting materials and intermediates in organic and organometallic chemistry[4,5] and α-haloketones are among the most popular chemical classes of affinity labels.[6]

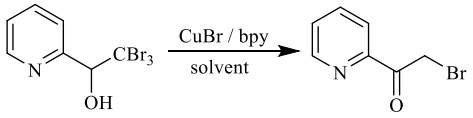
Hence the development of efficient methods for building α-halomethyl ketone motif has drawn considerable attention in organic synthesis. For example, Itoh and Glushkov groups respectively synthesized α-bromomethyl ketones from ethyl aromatic compounds by using bromination reagents like HBr or $Br_2$.[7,8] Chen's group obtained the α-monochlorinated ketones by using 1,3-dichloro-5,5-dimethylhydantoin (DCDMH), using silica gel as the catalyst, methanol as the solvent and heating for 1 h under reflux.[9] The group of Pravst have found that several aryl substituted ketones and cyclic ketones can be halogenated with *N*-halosuccinimides under solvent-free reaction conditions at various temperatures (20–80 °C).[10] Terent'ev's group also reported that acetophenone with different substituents produced α-bromomethyl ketones, α,α-dibromomethyl ketones and α,α,α-tribromomethyl ketones under $H_2O_2$-HBr system.[11] Patil and colleagues obtained α-bromoketones from olefins using bromide/bromate couple as nonhazardous brominating agent.[12] Zou and co-workers have developed the catalyzed hydration of haloalkynes in $Ce(SO_4)_2$/acid co-catalyzed system.[13] Ram group reported the reaction of α,α,α-trichloromethyl carbinols with different substituents to synthesize α-chloromethyl ketones in CuCl and bipyridine.[14] And other methods were also reported.[15]

However, among existing synthetic methods, these versatile reactions of synthesizing α-halomethyl ketones required the excess use of halogenating reagents, and harsh reaction conditions, and sometimes provided over chlorination, undesirable by-products. And by searching the documents, it is found that there are not many reports on the synthesis of α-bromoheterocyclic ketones by dehalogenation reaction. Herein, we report a novel and simple method for the synthesis of α-bromomethyl ketones with high regioselectivity.

Initially, we commenced our investigation by preparing α,α,α-tribromomethyl carbinols from aldehydes and bromoform with potassium hydroxide as the catalytic oxidizer.[16] Then oxidize α,α,α-tribromomethyl carbinols with PCC to obtain α,α,α-tribromomethyl ketones[17] and reduce it to get the target product. However, this synthetic route is complicated and produces by-products polluted the environment. Therefore, we decide to use the experimental method of the Ram group as a reference to directly synthesize α-bromomethyl ketones from α,α,α-tribromomethyl carbinols.[14]

We used α,α,α-tribromophenylethyl alcohol as the template reaction (Scheme 1) and investigated the effects of different reaction conditions (solvent, temperature, and the mass ratio of CuBr to bpy) on the synthesis of α-bromomethyl ketones (Table 1). At room temperature, we tried different solvents such as dichloromethane, toluene, ethanol, ether, and 1,2-dichloroethane (DCE). To our pleasure, the yield of the reaction was up to 30% when DCE was used as solvent (Table 1, entry 5). When the reaction temperature was raised to 90 °C (reflux), the yield was 50% (Table 1, entry 6). Then we changed the feed ratio of CuBr - bpy and found that the results were not satisfactory (Table 1, entries 7-9). Finally, we choose the entry 6 of Table 1 as the best condition for this reaction.

With the optimized conditions in hand, we next investigate the scope of reaction (Scheme 2), a series of α,α,α-tribromomethyl carbinols were utilized in optimal reaction condition (Table 2). When there is no substituent on the heterocyclic ring, the reaction offered moderate yields (Table 2, entries 2a, 2e). Whether there is electron-withdrawing or electron donating group on the heterocyclic ring, there is no obvious electron effect on thienone and pyridine products. Corresponding α-bromoheterocyclic ketones can be obtained with 40% ~ 54% yields (Table 2, entries 2a-2h). Then the aromatic compounds are tested and got the target product successfully. Aromatic α,α,α-tribromoethyl carbinols with a synthetically versatile -Cl, -Br, or -F substituent at the *otho*- or *para*-position all afforded the products in decent yields (Table 2, entries 2j, 2k, 2m). Substrates with both electron-donating and withdrawing groups could be transformed into the desired products in up to 81% yields (Table 2, entries 2n-2q). α,α,α-Tribromomethyl compounds were not as stable as α,α,α-trichloromethyl compounds, hence the yields were lower than theirs. These target products have been verified by $^{1}$H NMR and $^{13}$C NMR.



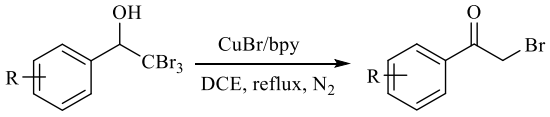

1a 2a

**Scheme 1**

**Table 1.** Optimization of reaction conditions for CuBr/bpy synthesis of α-bromomethyl ketones [a,b]

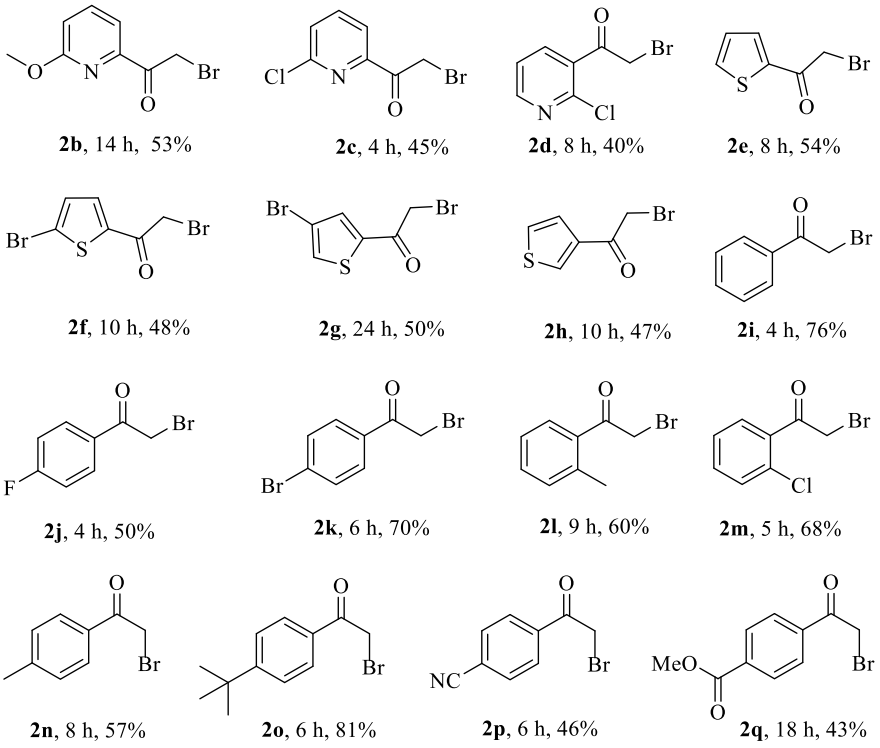
| Entry | solvent | CuBr (equiv.) | Bpy (equiv.) | Temp (°C) | Time (h) | yield[b] (%) |
|---|---|---|---|---|---|---|
| 1 | $CH_2Cl_2$ | 2.0 | 2.0 | rt | 24h | 25% |
| 2 | EtOH | 2.0 | 2.0 | rt | 24h | Trace |
| 3 | THF | 2.0 | 2.0 | rt | 24h | 10% |
| 4 | Ph-Me | 2.0 | 2.0 | rt | 24h | 18% |
| 5 | DCE | 2.2 | 2.2 | rt | 24h | 35% |
| **6** | **DCE** | **2.2** | **2.2** | **reflux** | **6h** | **50%** |
| 7 | DCE | 1.5 | 2.0 | reflux | 12h | 23% |
| 8 | DCE | 2.5 | 2.0 | reflux | 10h | 23% |
| 9 | DCE | 2.0 | 1.5 | reflux | 12h | 23% |

[a] α,α,α,-Tribromophenylethyl alcohol (0.2 mmol) added to CuBr/bpy (2.2 equiv.) system in refluxing DCE (10 mL). [b] Isolated yield.




**Scheme 2**

**Table 2.** Evaluation of α,α,α-tribromomethyl carbinol substrates [a,b]




[a] α,α,α,-Tribromomethyl carbinols (0.2 mmol) added to CuBr/bpy (2.2 equiv.) system in refluxing DCE (10 mL). [b] Isolated yield.

In conclusion, we describe a method for the synthesis of different substituent α-bromoheterocyclic ketones and α-bromomethyl ketones with simple operation. Notably, this transformation features abroad substrate scope, mild conditions, inexpensive and easily available raw materials and simple post-processing.

## EXPERIMENTAL

Visualization on TLC was achieved by use of UV light (254 nm) or iodine. NMR spectra were recorded on a Varian-400 MHz spectrometer (400 MHz $^{1}$H, 100 MHz $^{13}$C). The spectra were recorded in $CDCl_3$ as solvent at room temperature. $^{1}$H and $^{13}$C NMR chemical shifts are reported in ppm relative to either the residual solvent peak ($^{13}$C) (δ = 77.00 ppm) or TMS ($^{1}$H) (δ = 0 ppm) as an internal standard. Data for $^{1}$H NMR are reported as follows: chemical shift (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet, dd = doublet of doublet), integration, coupling constant (Hz) and assignment. Infrared spectra were recorded on a Nicolet MX-1E FT-IR spectrometer. HRMS spectra were recorded on a TOF-Q mass spectrometer.

First, put the bpy (0.44 mmol, 2.2 equiv.) in the reaction bottle and then dissolve in DCE (10 mL), to get the system 1. Then α,α,α-tribromomethyl carbinols (0.2 mmol, 1.0 equiv.) and CuBr (0.44 mmol, 2.2 equiv.) were added into the two-necked, flame-dried, round-bottomed flask and then dissolve in DCE (10 mL) to get the system 2. Using the round bottom flask equipped with spherical condensing tube, the system 1 was dripped into the system 2, then the mixture was heated at reflux with stirring by magnetic force for hours. As the reaction proceeded, the color of the mixture in the flask will change from dark green to brown. And the progress of the reaction was monitored by TLC. After reaction, stop heating and cool for 10 min, the system needs to be filtered by vacuum extraction, washed with EtOAc for many times and then solvents such as EtOAc concentrated by rotary evaporation. Finally it should be purified by silica gel column using a solution of 10% EtOAc in petroleum ether as the solvent for elution to give pure **2a-2q**.

**2-Bromo-1-(pyridin-2-yl)ethan-1-one (2a)**[15]: yield 20 mg (50%), colorless oil. IR (in KBr) 3057, 2923, 2852, 1712, 1582, 1437, 1395, 1309, 1202, 997, 772, 614 cm$^{-1}$; $^{1}$H NMR (400 MHz, $CDCl_3$), δ, ppm: 4.83 (1H, s), 5.09 (1H, s), 7.48-7.52 (1H, m), 7.82-7.87 (1H, m), 8.08 (1H, d, *J* = 8.0 Hz), 8.64-8.67 (1H, t, *J* = 6.4 Hz); $^{13}$C NMR (100 MHz, $CDCl_3$), δ, ppm: 32.38, 122.34-122.68 (d, *J* = 34.0 Hz), 127.81-127.93 (d, *J* = 12.0 Hz), 137.16-137.18 (d, *J* = 2.0 Hz), 149.10-149.13 (d, *J* = 3.0 Hz), 151.35, 192.46

**2-Bromo-1-(6-methoxypyridin-2-yl)ethan-1-one (2b)**: yield 24.4 mg (53%), yellow oil. IR (in KBr) 3082, 2950, 2852, 2619, 1715, 1597, 1472, 1346, 1282, 1202, 1037, 988, 905, 807, 732, 634 cm$^{-1}$; $^{1}$H NMR (400 MHz, $CDCl_3$), δ, ppm: 3.96 (3H, d, *J* = 5.2 Hz), 4.77 (2H, s), 6.96-6.98 (1H, *J* = 7.6 Hz),

7.68-7.74 (2H, m); $^{13}$C NMR (100 MHz, $CDCl_3$), δ, ppm: 32.37, 53.56-53.58 (d, *J* = 2.0 Hz), 115.72, 116.05-116.42 (m), 138.34-138.36 (d, *J* = 2.0 Hz), 148.91, 163.34, 191.79; ESI HRMS *m/z* calcd. for $C_8H_8BrNO_2$ $[M]^+$ 228.97384, found 228.97328.

**2-Bromo-1-(6-chloropyridin-2-yl)ethan-1-one (2c)**: yield 21.1 mg (45%), yellow oil. IR (in KBr) 3079, 2924, 2851, 1715, 1566, 1432, 1308, 1205, 1146, 996, 802, 706, 630 $cm^{-1}$; $^1$H NMR (400 MHz, $CDCl_3$), δ, ppm: 4.79 (2H, s), 7.53 (1H, d, *J* = 6.8 Hz), 7.81 (1H, t, *J* = 8.0 Hz), 7.99 (1H, d, *J* = 7.6 Hz); $^{13}$C NMR (100 MHz, $CDCl_3$), δ, ppm: 32.08, 120.90-121.22 (d, *J* = 32.0 Hz), 128.87-129.00 (d, *J* = 13.0 Hz), 139.80-139.83 (d, *J* = 3.0 Hz), 151.43, 151.70, 191.08; ESI HRMS *m/z* calcd. for $C_7H_5BrClNO$ $[M]^+$ 232.92430, found 232.92370.

**2-Bromo-1-(2-chloropyridin-3-yl)ethan-1-one (2d)**: yield 18.7 mg (40%), yellow oil. IR (in KBr) 2923, 1704, 1572, 1398, 1284, 1088, 993, 808, 668 $cm^{-1}$; $^1$H NMR (400 MHz, $CDCl_3$), δ, ppm: 4.53 (2H, s), 7.35-7.38 (1H, m), 7.90 (1H, dd, *J* = 2.0, 8.0 Hz), 8.50-8.52 (1H, m); $^{13}$C NMR (100 MHz, $CDCl_3$), δ, ppm: 33.77, 122.64, 132.66, 139.53, 147.32, 152.04, 192.86; ESI HRMS *m/z* calcd. for $C_7H_5BrClNO$ $[M]^+$ 232.92430, found 232.92377.

**2-Bromo-1-(thiophen-2-yl)ethan-1-one (2e)**[13]: yield 22.1 mg (54%), colorless oil. IR (in KBr) 3013, 2918, 2850, 2378, 2320, 1673, 1515, 1410, 1354, 1279, 1181, 1065, 860, 728 $cm^{-1}$; $^1$H NMR (400 MHz, $CDCl_3$), δ, ppm: 4.37 (2H, s), 7.18 (1H, s), 7.73 (1H, s), 7.82 (1H, s); $^{13}$C NMR (100 MHz, $CDCl_3$), δ, ppm: 30.57, 128.38, 133.50, 135.21, 140.73, 184.37

**2-Bromo-1-(5-bromothiophen-2-yl)ethan-1-one (2f)**[8b]: yield 27.2 mg (48%), yellow solid, mp 67-68 °C. IR (in KBr) 3084, 2997, 2951, 1670, 1518, 1406, 1319, 1216, 983, 795 $cm^{-1}$; $^1$H NMR (400 MHz, $CDCl_3$), δ, ppm: 4.26 (2H, s), 7.11 (1H, dd, *J* = 1.6, 4.0 Hz), 7.52 (1H, dd, *J* =0.8, 4.0 Hz); $^{13}$C NMR (100 MHz, $CDCl_3$), δ, ppm: 29.72, 124.50, 131.53, 133.75, 142.11, 183.39; ESI HRMS *m/z* calcd. for $C_6H_4Br_2OS$ $[M]^+$ 281.83496, found 281.83454.

**2-Bromo-1-(4-bromothiophen-2-yl)ethan-1-one (2g)**: yield 28.4 mg (50%), yellow oil, IR (in KBr) 3100, 2928, 2851, 1667, 1508, 1394, 1340, 1283, 1185, 823, 636 $cm^{-1}$; $^1$H NMR (400 MHz, $CDCl_3$), δ, ppm: 4.29 (2H, s), 7.59 (1H, s), 7.68 (1H, s); $^{13}$C NMR (100 MHz, $CDCl_3$), δ, ppm: 29.91, 111.14, 132.36, 135.43, 141.05, 183.49; ESI HRMS *m/z* calcd. for $C_6H_4Br_2OS$ $[M]^+$ 281.83496, found 281.83451.

**2-Bromo-1-(thiophen-3-yl)ethan-1-one (2h)**[13]: yield 19.2 mg (47%), yellow oil, IR (in KBr) 3097, 2857, 1723, 1663, 1515, 1412, 1356, 1288, 1198, 1063, 941, 856, 728, 620 $cm^{-1}$; $^1$H NMR (400 MHz, $CDCl_3$) δ, ppm: 4.32 (2H, s), 7.33 (1H, d, *J* = 2.8 Hz), 7.55 (1H, d, *J* = 5.2 Hz), 8.16 (1H, s); $^{13}$C NMR (100 MHz, $CDCl_3$), δ, ppm: 28.96, 111.13, 132.40, 135.43, 141.02, 183.50

**2-Bromo-1-phenylethan-1-one (2i)**[13]: yield 30.3 mg (76%), white solid, mp 45-46 °C. IR (in KBr) 3064, 3015, 2975, 2850, 1697, 1579, 1418, 1270, 1192, 1161, 981, 801, 782, 684 $cm^{-1}$; $^1$H NMR (400 MHz, $CDCl_3$), δ, ppm: 4.46 (2H, s), 7.50 (2H, t, *J* = 8.0 Hz), 7.63~7.59 (1H, m), 8.00 (2H, d, *J* = 8.0 Hz); $^{13}$C

NMR (100 MHz, $CDCl_3$), δ, ppm: 30.90, 128.85 (2C), 128.92 (2C), 133.94 (2C), 191.25

**2-Bromo-1-(4-fluorophenyl)ethan-1-one (2j)**[13]: yield 21.7 mg (50%), white solid, mp 43-44 °C. IR (in KBr) 2924, 2853, 2360, 1694, 1596, 1507, 1393, 1198, 997, 825 $cm^{-1}$; $^1H$ NMR (400 MHz, $CDCl_3$), δ, ppm: 4.41 (2H, s), 7.20-7.14 (2H, m), 8.04~8.00 (2H, m); $^{13}C$ NMR (100 MHz, $CDCl_3$), δ, ppm: 39.31, 116.19-115.97 (2C, d, *J* = 22.0 Hz), 130.34-130.31 (2C, d, *J* = 3.0 Hz), 131.76-131.67 (d, *J* = 9.0 Hz), 167.41-164.86 (d, *J* = 255.0 Hz), 189.80

**2-Bromo-1-(4-chlorophenyl)ethan-1-one (2k)**[13]: yield 38.9 mg (70%), white solid, mp 109-110 °C. IR (in KBr) 2998, 2953, 2852, 1694, 1584, 1397, 1197, 1071, 990, 808 $cm^{-1}$; $^1H$ NMR (400 MHz, $CDCl_3$), δ, ppm: 4.40 (2H, s), 7.65 (2H, d, *J* = 8.0 Hz), 7.86 (2H, d, *J* = 8.0 Hz); $^{13}C$ NMR (100 MHz, $CDCl_3$), δ, ppm: 30.33, 129.29 (2C), 130.40 (2C), 132.21, 132.62, 190.38

**2-Bromo-1-(*o*-tolyl)ethan-1-one (2l)**[15b]: yield 25.5 mg (60%), colorless oil. IR (in KBr) 2918, 2849, 2342, 1679, 1296, 1183, 1101, 850 $cm^{-1}$; $^1H$ NMR (400 MHz, $CDCl_3$), δ, ppm: 2.53 (3H, s), 4.43 (2H, s), 7.31 (2H, d, *J* = 12.0 Hz), 7.43 (1H, t, *J* = 8.0 Hz,), 7.68 (1H, d, *J* = 8.0 Hz); $^{13}C$ NMR (100 MHz, $CDCl_3$), δ, ppm: 21.41, 33.68, 125.78, 128.98, 132.31, 132.33, 134.43, 139.71, 194.16

**2-Bromo-1-(2-chlorophenyl)ethan-1-one (2m)**[15]: yield 31.7 mg (68%), colorless oil. IR (in KBr) 3026, 2858, 2359, 2432, 1713, 1681, 1420, 1262, 873, 714 $cm^{-1}$; $^1H$ NMR (400 MHz, $CDCl_3$), δ, ppm: 4.52 (2H, s), 7.39~7.35 (1H, m), 7.45 (2H, d, *J* = 4.0 Hz), 7.68 (1H, d, *J* = 8.0 Hz); $^{13}C$ NMR (100 MHz, $CDCl_3$), δ, ppm: 34.48, 127.12, 130.23, 130.56, 131.29, 132.72, 136.21, 193.98

**2-Bromo-1-(*p*-tolyl)ethan-1-one (2n)**[13]: yield 24.3 mg (57%), white solid, mp 49-50 °C. IR (in KBr) 2952, 2918, 2360, 1692, 1606, 1392, 1284, 1196, 998, 878 $cm^{-1}$; $^1H$ NMR (400 MHz, $CDCl_3$), δ, ppm: 2.43 (3H, s), 4.43 (2H, s), 7.30 (2H, d, *J* = 8.0 Hz), 7.90 (2H, d, *J* = 8.0 Hz,); $^{13}C$ NMR (100 MHz, $CDCl_3$), δ, ppm: 21.75, 30.91, 129.04 (2C), 129.54 (2C), 131.46, 145.00, 190.95

**2-Bromo-1-(4-(*tert*-butyl)phenyl)ethan-1-one (2o)**[13]: yield 39.3 mg (81%), colorless oil. IR (in KBr) 2964, 2905, 2869, 1697, 1676, 1603, 1564, 1462, 1408, 1364, 1271, 1194, 1107, 982, 850 $cm^{-1}$; $^1H$ NMR (400 MHz, $CDCl_3$), δ, ppm: 1.35 (9H, s), 4.44 (2H, s), 7.52 (2H, d, *J* = 8.0 Hz), 7.93 (2H, t, *J* = 4.0 Hz); $^{13}C$ NMR (100 MHz, $CDCl_3$), δ, ppm: 30.89 (3C), 31.01, 35.25, 125.82 (2C), 128.91 (2C), 131.36, 157.89, 190.90

**4-(2-Bromoacetyl)benzonitrile (2p)**[13]: yield 20.6 mg (46%), white solid, mp 91-92 °C IR (in KBr) 3090, 2992, 2225, 1707, 1566, 1404, 1291, 1193, 994, 828 $cm^{-1}$; $^1H$ NMR (400 MHz, $CDCl_3$), δ, ppm: 4.43 (2H, s), 7.81 (2H, d, *J* = 8.0 Hz), 8.09 (2H, d, *J* = 8.0 Hz); $^{13}C$ NMR (100 MHz, $CDCl_3$), δ, ppm: 30.01, 117.15, 117.63, 129.37 (2C), 132.66 (2C), 136.87, 190.06

**Methyl 4-(2-bromoacetyl)benzoate (2q)**[13]: yield 22.1 mg (43%), white solid, mp 135-136 °C. IR (in KBr) 2959, 2360, 1675, 1590, 1484, 1396, 1277, 1166, 1011, 870, 811 $cm^{-1}$; $^1H$ NMR (400 MHz, $CDCl_3$), δ, ppm: 3.95 (3H, s), 4.48 (2H, s), 8.04 (2H, d, *J* = 8.0 Hz), 8.15 (2H, d, *J* = 8.0 Hz); $^{13}C$ NMR (100 MHz,

$CDCl_3$), δ, ppm: 30.67, 52.55, 128.84 (2C), 129.98 (2C), 134.59, 137.11, 165.91, 190.79

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