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Facile synthesis of 5-bromotropono[c]-fused pyrazoles and isoxazole

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Abstract: A facile synthesis of a series of new 5-bromotropono[c]pyrazole derivatives (**3** and **10**–**15**) as well as 5-bromotropono[c]isoxazole (**17**) is described, involving a condensation reaction of 3-acetyl-5-bromotropolone (**1**) with hydrazine monohydrate (**2**), arylhydrazine hydrochlorides (**4**–**9**), and hydroxylamine hydrochloride (**16**), respectively. All the synthesized compounds were obtained in good yields of 56%–77% and their structures were characterized by spectral data and HRMS.

Key words: Tropono[c]pyrazole, tropono[c]isoxazole, condensation reaction, hydrazine monohydrate, arylhydrazine, hydroxylamine

1. Introduction

The pyrazole skeleton often appears as an important structural component in both biologically active and natural compounds exhibiting a wide range of pharmacological properties.[1,2] In particular, the important role of ring-fused pyrazoles in medicinal and pharmaceutical chemistry is indisputable and well reflected by a large number of recent publications,[3–6] including some excellent reviews.[7,8] Most fused pyrazole compounds reported in the literature comprise a common heterocyclic ring moiety, such as pyrazolopyrimidine,[9] pyrazolopyridine,[10] pyrazoloquinoline,[11] pyrazoloindol,[12] benzofuropyrazole,[13] benzopyranopyrazole,[14] and synthetic analogues thereof. In addition, as is well known, the ring-fused isoxazole skeleton is also an important structural unit that can be found in various bioactive compounds such as those with anti-HIV,[15] antifungal,[16] and nematicidal properties.[17] Accordingly, much work has been directed toward the design and synthesis of various ring-fused isoxazole derivatives.[18–20]

It is worth mentioning that a combination of a heterocycle moiety fused with a tropone ring may increase their biological activities or create new medicinal properties due to the different electronic distribution and the additional basic character of the tropone ring.[21–23] For example, structural variations of established drugs with the tropone ring resulted in enhanced anticancer activity.[23] In addition, some natural products and synthetic compounds containing heterocyclic-fused tropone moiety exhibit potent biological and pharmacological activities such as antitumor and antimalarial activities.[24–26] Therefore, there is much current interest in assembling a tropone ring by fusing with heterocyclic systems.[27,28] However, there are very few examples in the literature concerning the synthesis of tropone-fused pyrazoles or isoxazole,[29] although such compounds are attractive in the field of new drug discovery.

On the basis of these observations, and in view of structural diversity playing a prominent role in

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medicinal and combinatorial chemistry for a faster and more efficient route towards new drug discovery,[30] the synthesis of novel tropone-fused pyrazoles as well as isoxazole is not only synthetically challenging but also potentially biologically interesting. Therefore, in the context of our ongoing studies concerning the preparation of potential biologically active heterocycles,[31–33] and in diversifying our work on the synthesis of new tropone compounds,[34–36] we wish to report herein a simple and efficient protocol for the synthesis of structurally novel tropone-fused pyrazoles and isoxazole, namely 1-aryl-5-bromo-3-methyltropono[*c*]pyrazoles and 5-bromo-3- methyl-tropono[*d*]isoxazole.

2. Experimental

2.1. Materials and reagents

The chemicals used in this work were obtained from Fluka and were used without purification. The melting points were determined by using a WRS-1B melting point apparatus and were uncorrected. The IR spectra of the compounds in KBr pellets were obtained in the range of 400–4000 cm^{-1} on a Shimadzu FTIR-8400S spectrophotometer. ^{1}H NMR was measured with a BRUKER BRX 400 at 400 MHz using $CDCl_3$ or DMSO-d_6 as the solvent. The reported chemical shifts (δ values) are given in parts per million downfield from tetramethylsilane (TMS) as the internal standard. HRMS (ESI) data were acquired on a Bruker Customer micrOTOF-Q 125 high-resolution mass spectrometer with ESI. The progress of reactions was monitored by thin-layer chromatography (TLC) on silica gel GF254 using ethyl acetate/petroleum ether (1:3) as eluent.

2.2. General procedure for the synthesis of 5-bromo-3-methyltropono[*c*]pyrazole derivatives (3 and 10–15)

To a stirred solution of 3-acetyl-5-bromotropolone **1** (0.24 g, 1 mmol) in 10 mL of MeOH was added the respective hydrazine monohydrate (80%) (**2**) and arylhydrazine hydrochlorides (2 mmol) (**4**–**9**). The resulting mixture was heated at reflux temperature for 12 h. After the reaction was complete (TLC), the mixture was cooled to room temperature followed by addition of 10 mL of water to it. The resulting precipitate was collected by filtration and purified by recrystallization from ethanol to give products **3** and **10**–**15** in 56%–77% yields.

5-Bromo-3-methyltropono[*c*]pyrazole (3)

This compound was obtained as a white solid, yield 72%, mp 142–144 °C. IR (KBr): 3139 (NH), 1620 (C=O), 1584 (C=N), 1533, 1510, 1416, 1400, 1331, 1228, 1214, 1125, 1013 cm^{-1}; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 2.56 (s, 3H, Me), 6.89 (d, 1H, J = 10.5 Hz, Tropone-H), 7.41 (dd, 1H, J = 10.5, 1.5 Hz, Tropone-H), 7.84 (d, 1H, J = 1.5 Hz, Tropone-H), 13.67 (br s, 1H, NH). HRMS (ESI): m/z $[M+Na]^+$ Calcd for $C_9H_7^{79}BrN_2NaO^+$: 260.9632. Found: 260.9636.

5-Bromo-3-methyl-1-phenyltropono[*c*]pyrazole (10)

This compound was obtained as a yellow solid, yield 77%, mp 183–184 °C. IR (KBr): 1626 (C=O), 1591 (C=N), 1548, 1497, 1460, 1389, 1337, 1205, 1146, 1075, 1008 cm^{-1}; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 2.59 (s, 3H, Me), 6.77 (d, 1H, J = 10.6 Hz, Tropone-H), 7.34–7.36 (m, 2H, ArH), 7.43–7.47 (m, 4H, ArH), 7.81 (d, 1H, J = 1.5 Hz, Tropone-H). HRMS (ESI): m/z $[M+Na]^+$ Calcd for $C_{15}H_{11}^{79}BrN_2NaO^+$: 336.9945. Found: 336.9936.

5-Bromo-1-(4-methoxyphenyl)-3-methyltropono[*c*]pyrazole (11)

This compound was obtained as colorless crystals, yield 75%, mp 194–195 °C. IR (KBr): 1628 (C=O),

1593 (C=N), 1546, 1511, 1448, 1403, 1328, 1304, 1266, 1177, 1069, 1029 cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz): δ 2.58 (s, 3H, Me), 3.86 (s, 3H, OMe), 6.76 (d, 1H, J = 10.5 Hz, Tropone-H), 6.96 (d, J = 8.4 Hz, 2H, Ph-H), 7.28 (d, J = 8.4 Hz, 2H, Ph-H), 7.42 (dd, 1H, J = 10.5, 1.6 Hz, Tropone-H), 7.80 (d, 1H, J = 1.5 Hz, Tropone-H). HRMS (ESI): m/z $[M+Na]^+$ Calcd for $C_{16}H_{13}^{79}BrN_2NaO_2^+$: 367.0051. Found: 367.0043.

5-Bromo-1-(4-chlorophenyl)-3-methyltropono[*c*]pyrazole (12)

This compound was obtained as yellow crystals, yield 74%, mp 251–252 °C. IR (KBr): 1621 (C=O), 1589 (C=N), 1561, 1497, 1461, 1400, 1331, 1252, 1204, 1137, 1088, 1004 cm^{-1}; ^1H NMR (DMSO-d_6, 400 MHz): δ 2.58 (s, 3H, Me), 6.77 (d, 1H, J = 10.5 Hz, Tropone-H), 7.29 (d, J = 8.4 Hz, 2H, Ph-H), 7.42 (d, J = 8.4 Hz, 2H, Ph-H), 7.45 (dd, 1H, J = 10.5, 1.5 Hz, Tropone-H), 7.81 (d, 1H, J = 1.5 Hz, Tropone-H). HRMS (ESI): m/z $[M+Na]^+$ Calcd for $C_{15}H_{10}^{79}Br^{35}ClN_2NaO^+$: 370.9555. Found: 370.9548.

5-Bromo-1-(4-bromophenyl)-3-methyltropono[*c*]pyrazole (13)

This compound was obtained as a yellow solid, yield 72%, mp 259–260 °C. IR (KBr): 1628 (C=O), 1590 (C=N), 1498, 1452, 1400, 1332, 1256, 1207, 1064, 1005 cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz): δ 2.58 (s, 3H, Me), 6.78 (d, 1H, J = 10.5 Hz, Tropone-H), 7.25 (d, J = 7.8 Hz, 2H, Ph-H), 7.45 (dd, 1H, J = 10.5, 1.5 Hz, Tropone-H), 7.58 (d, J = 7.8 Hz, 2H, Ph-H), 7.80 (d, 1H, J = 1.5 Hz, Tropone-H). HRMS (ESI): $m/z[M+H]^+$ HRMS (ESI): m/z $[M+Na]^+$ Calcd for $C_{15}H_{10}^{79}Br_2N_2NaO^+$: 414.9050. Found: 414.9057.

5-Bromo-1-(4-cyanotrophenyl)-3-methyltropono[*c*]pyrazole (14)

This compound was obtained as an orange solid, yield 60%, mp 188–190 °C. IR (KBr): 2221 (C≡N), 1651 (C=O), 1598 (C=N), 1574, 1561, 1518, 1492, 1397, 1325, 1257, 1220, 1139, 1089, 1042 cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz): δ 2.66 (s, 3H, Me), 6.81 (d, 1H, J = 10.5 Hz, Tropone-H), 7.46 (d, 2H, J = 7.8 Hz, Ph-H), 7.69 (dd, 1H, J = 10.5, 1.6 Hz, Tropone-H), 7.62 (d, J = 7.8 Hz, 2H, Ph-H), 7.84 (d, 1H, J = 1.5 Hz, Tropone-H), 8.38 (d, 1H, J = 2.0 Hz, Ph-H). HRMS (ESI): m/z $[M+Na]^+$ Calcd for $C_{16}H_{10}^{79}BrN_3NaO^+$: 361.9897. Found 361.9889.

5-Bromo-1-(2,4-dinitrophenyl)-3-methyltropono[*c*]pyrazole (15)

This compound was obtained as a yellow solid, yield 56%, mp 194–196 °C. IR (KBr): 1647 (C=O), 1609, 1585 (C=N), 1554, 1513, 1484, 1402, 1338, 1260, 1214, 1147, 1097, 1038 cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz): δ 2.62 (s, 3H, Me), 6.77 (d, 1H, J = 10.5 Hz, Tropone-H), 7.55 (dd, 1H, J = 10.5, 1.6 Hz, Tropone-H), 7.76 (d, 1H, J = 7.8 Hz, Ph-H), 7.87 (d, 1H, J = 1.5 Hz, Tropone-H), 8.56 (dd, 1H, J = 7.8, 2.0 Hz, Ph-H), 9.02 (d, 1H, J = 2.0 Hz, Ph-H). HRMS (ESI): m/z $[M+Na]^+$ Calcd for $C_{15}H_9^{79}BrN_4NaO_5^+$: 426.9646. Found 426.9652.

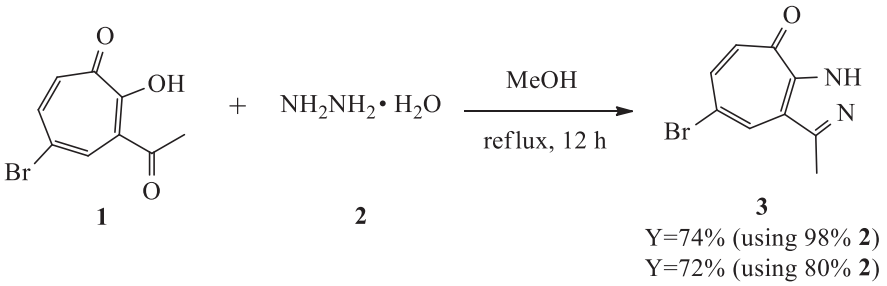
2.3. Procedure for the synthesis of 5-bromo-3-methyl-8*H*-tropono[*d*]isoxazole (17)

To a stirred solution of 3-acetyl-5-bromotropolone **1** (0.24 g, 1 mmol) in 10 mL of MeOH was added hydroxylamine hydrochloride (**16**) (0.14 g, 2 mmol). The resulting mixture was heated at reflux for 10 h. After the reaction was complete (TLC), the mixture was cooled to room temperature and 10 mL of water was added to it. The resulting precipitate was collected by filtration and purified by recrystallization from ethanol to give product **17**. This compound was obtained as a brown solid, yield 70%, mp 164–166 °C. IR (KBr): 1624 (C=O), 1578, 1519, 1403, 1325, 1252, 1219, 1132, 1023 cm^{-1}; ^1H NMR (DMSO-d_6, 400 MHz): δ 2.60 (s, 3H, Me), 7.08 (d, 1H, J = 10.5 Hz, Tropone-H), 7.63 (dd, 1H, J = 10.5, 1.5 Hz, Tropone-H), 7.65 (d, 1H, J = 1.4 Hz, Tropone-H). HRMS (ESI): m/z $[M+Na]^+$ Calcd for $C_9H_6^{79}BrNNaO_2^+$: 261.9472. Found 261.9467.

3. Results and discussion

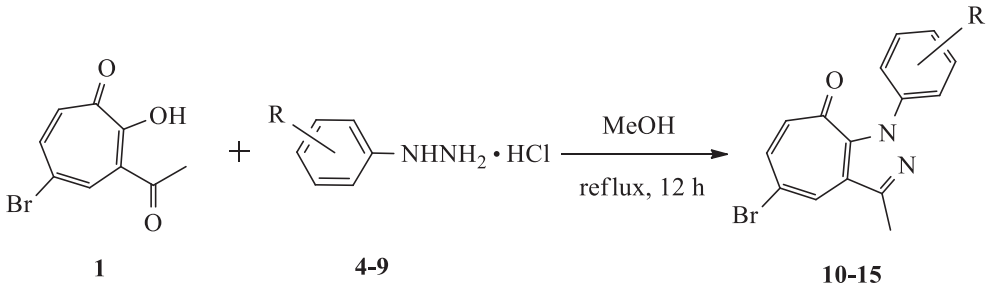
As far as we know, 3-acetyl-5-bromotropolone (**1**) has been synthesized conveniently for many years, but the further modification of it was very limited.[37] In this regard, we have reported the synthesis of a series of flavonoid-like troponoids employing 3-acetyl-5-bromotropolone (**1**) as the starting compound.[38] In the context of our ongoing studies on troponoid chemistry and further extending the diversity of our previous work, we have become interested in the synthesis of new 5-bromo-tropono[*c*]pyrazole derivatives by employing **1** for current medicinal chemistry needs.

Initially, we investigated the condensation reaction of **1** with hydrazine monohydrate (**2**) by refluxing **1** and 2.0 equiv. of hydrazine monohydrate (98%) in methanol (10 mL) as shown in Scheme 1. After the reaction was completed as monitored by TLC, the desired product **3** was obtained in a good yield of 74%. Further, when conducting the reaction using relatively cheap hydrazine monohydrate (80%) (Scheme 1), we were delighted to find that the reaction also proceeded well and the product was obtained in a comparable yield of 72%.



Scheme 1. Synthesis of 5-bromo-3-methyltropono[*c*]pyrazole (**3**).

Subsequently, we examined the reaction of **1** with some free arylhydrazines (**4**–**9**) (2.0 equiv.) under the same reaction conditions. However, we found that the condensation reaction proceeded poorly, and the desired products were obtained in low yields of 21%–48% even after 48 h. It is worth mentioning that the observation in our case was very similar to that reported by Lee et al., where the use of free arylhydrazines also gave products in poor yields.[39] Considering these results, we attempted to use the corresponding arylhydrazine hydrochloride as the reaction partner as shown in Scheme 2.



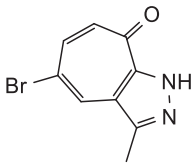
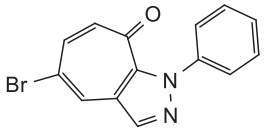
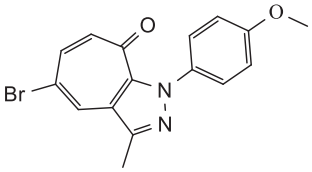
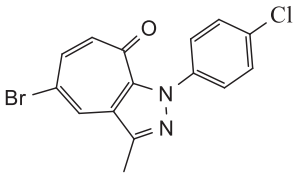
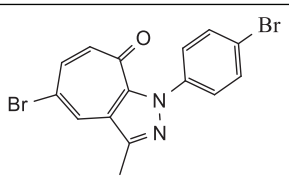
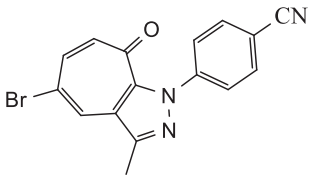
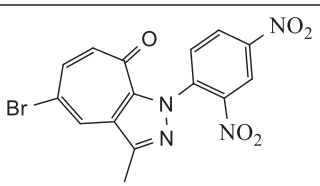
Scheme 2. Synthesis of 1-aryl-5-bromo-3-methyltropono[*c*]pyrazoles (**10**–**15**).

To our delight, an improvement in terms of yields and reaction time was achieved, and the desired 1-aryl-5-bromo-3-methyltropono[*c*]pyrazoles (**10**–**15**) were obtained in satisfactory yields of 56%–77% after 12 h. The reaction results are summarized in the Table.

As shown in the Table, all the desired products were obtained in satisfactory yields except for compounds **14** and **15** (entries 6 and 7). The products **14** and **15** were obtained in moderate yields of 60% and 52%,

respectively, which we attributed to the strong electron-withdrawing effects of the CN and NO_2 groups, which rendered the condensation reaction unfavorable.

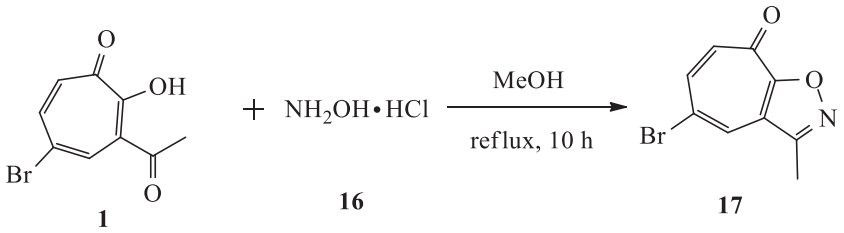
Table. Yields of the newly synthesized 5-bromo-tropono[*c*]pyrazoles (**3**, **10**–**15**).

Entry	Product	**3, 10 -15**	Yield[a]/%
1		**3**	72
2		**10**	77
3		**11**	75
4		**12**	74
5		**13**	72
6		**14**	60
7		**15**	52

[a] Isolated yield.

Additionally, in diversifying our work on new tropone-fused heterocyles, the same reaction conditions were further applied to the reaction with hydroxylamine hydrochloride (**16**) with the aim of constructing a novel tropone-fused isoxazole system as shown in Scheme 3. Interestingly, hydroxylamine hydrochloride was

equally amenable to the conditions, only varying the reaction time according to TLC monitoring, and the corresponding 5-bromo-3-methyl-tropono[*d*]isoxazole (**17**) was obtained in a comparable yield of 70%.



Scheme 3. Synthesis of 5-bromo-3-methyltropono[*d*]isoxazole (**17**).

To the best of our knowledge, none of the newly synthesized compounds **3**, **10**–**15**, and **17** have yet been reported and their structures were easily established based on spectral data and HRMS. As an example, the main features of the ^{1}H NMR data of compound **10** showed the absence of the signal belonging to OH moiety of the precursor, along with the signals for 8 aromatic protons exactly matching its structure in the range of the aromatic region of 6.77–7.81 ppm. Further, its molecular formula was established to be $C_{15}H_{11}BrN_2O$ in accordance with the suggested molecular structure by its HRMS spectrum, which showed a *pseudo*-molecular-ion peak at m/z 336.9936 ([M+Na]$^+$; calc. for $C_{15}H_{11}^{79}BrN_2NaO^+$: 336.9945), indicating the presence of 11° of unsaturation.

4. Conclusions

We have achieved a facile synthesis of previously unattainable 5-bromo-tropono[*c*]pyrazole derivatives. These compounds could be potentially applied for the development of biologically and pharmaceutically important drugs. In addition, it is important to mention that these newly synthesized compounds contain a derivatizable bromo group on the tropone ring, which makes them particularly appealing, since the functional group provides ample opportunity for further synthetic manipulation, for example, by cross coupling reactions to obtain more complex compounds. Further elaboration of these compounds to a variety of other functional groups is ongoing and will be covered in further publications.

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