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# Two-Step Syntheses of 3-Methyl and 3-Phenyl-1,2,4-Benzotriazines

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ABSTRACT: *3-Methyl-1,2,4-benzotriazine and some of its derivatives were prepared in moderate yields (50–70%) via a reductive cyclization by a $PtO_2$-catalyzed hydrogenation of the corresponding 2-nitrophenylhydrazones of the pyruvic acid. The latter compounds were obtained in yields higher than 90% by reacting 2-nitrophenylhydrazines with sodium pyruvate salt. Three 3-phenyl-1,2,4-benzotriazine compounds were also produced via a reductive cyclization by a Pt/C-catalyzed hydrogenation of their corresponding 2-nitrophenylhydrazono-ethers in high yields (>70%).* © 2006 Wiley Periodicals, Inc. Heteroatom Chem 17:166–172, 2006; Published online in Wiley InterScience (www.interscience.wiley.com). DOI 10.1002/hc.20200

## *INTRODUCTION*

The reductive cyclization of nitrophenylhydrazones has been widely used to afford nitrogen-based heterocyclic molecules. To name but a few, there are 4*H*-pyrazolo-[1,5-*a*]benzimidazoles [1], ethyl-(1,2,4-benzotriazine-3-yl)acetate and its derivatives [2], benzo[1,2-*b*:5,4-*b*′]bis(1*H*)-imidazo[1,2-*b*]pyrazoles [3], 1,2-dihydro-1,2,5-benzotriazepines [4], and 3,4-dihydropyridazino[1,6-*a*]benzoimidazoles [5]. Several pathways for the synthesis of 1,2,4-benzotriazines have been reported [6–12]. For example, 3-amino-1,2,4-benzotriazine was prepared in 57% yield by the reduction of 3-amino-1,2,4-benzotriazine 1-oxide with sodium dithionite, the latter oxide was obtained in 39% yield by reacting nitroaniline with cyanamide [13]. Various pathways for the synthesis of 3-phenyl-1,2,4-benzotriazine have been described [9,12,14–19]. It is enlightening to recall that the oxidized of 1,2,4,benzotriazines have been of special interest because of their potential antitumor activity [20,21]. Indeed, the synthesis of 3-amino-1,2,4-benzotriazine-1,4-oxide, also known as tirapazamine, is nowadays a central occupation for many workers [22–24]. Accordingly, 1,2,4-benzotriazines are valuable precursors to very promising cancer therapeutic molecules.

In continuation of our work on heterocyclic synthesis [5], we developed short pathways to produce 3-methyl and 3-phenyl-1,2,4-benzotriazines from phenylhydrazones and phenylhydrazono-ethers, respectively. The hydrazones have been used largely as starting materials in the Fischer' indole synthesis [25].

## *EXPERIMENTAL*

Apart from the starting material for the synthesis of **1a** and **3a**, which was purchased from Aldrich, the remaining phenylhydrazines were synthesized

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according to the method described in the literature [26]. Benzonitrile, sodium pyruvate, perchloric acid, ethanol, $PtO_2$, Pt/C, methanol, and chloroform $CDCl_3$ were purchased from Aldrich. The iminoester used in Eq. (3) was prepared from benzonitrile using the Pinner reaction.

Melting points were recorded with a Boetus microhotstage. Elemental analyses were performed on a HERAEUS CHN-O-RAPID analyzer. Infrared and UV–Vis spectra were measured with a Specord M80 Carl Zeiss Jena spectrophotometer and a Beckmann DU 650 spectrophotometer, respectively. $^1$H NMR spectra were recorded using a Varian Gemini 200 spectrometer; chemical shifts for NMR signals are reported in ppm from TMS (tetramethylsilane) used as an internal standard.

### *General Procedure for Synthesis of* **1a–f**

Sodium pyruvate (1.1 g, 0.01 mol) and the corresponding 2-nitrophenylhydrazine (0.01 mol) were dissolved in 10 mL of ethanol and the mixture was stirred in the presence of 10 mL of a 35% ethanolic solution of perchloric acid. Immediately, a yellow crystal precipitated and was sequentially filtered, washed with water until neutral pH, and dried. Finally, the product was purified by recrystallization; compounds **1a** and **1b–f** were recrystallized from toluene and ethanol, respectively.

### *General Procedure for Synthesis of* **2a–f**

The corresponding 2-nitrophenylhydrazone of pyruvic acid (3 mmol) dissolved in 50 mL of ethanol was hydrogenated in the presence of $PtO_2$ at room temperature; about 202 mL of hydrogen was absorbed before the end of the reaction. Afterwards, the catalyst was removed by filtration and the solvent was distilled off. The remaining yellow crystals were recrystallized; **2a, 2b, 2d**, and **2e** were recrystallized from petroleum ether and, **2c** and **2f** from ethanol and methanol, respectively.

### *General Procedure for Synthesis of* **4a–c**

The corresponding nitrophenylhydrazine (0.01 mol) and 0.01 mol of the imino-ester (1.49 g) were dissolved in 25 mL of ethanol and the mixture was stirred at room temperature for 1 h. The obtained yellow crystals (**3a–c**) were filtered and dried. The latter substance was dissolved in 25 mL of ethanol and subjected to reduction by hydrogen in the presence of Pt/C at room temperature. Afterwards, the catalyst was removed by filtration and the solvent was distilled off. The obtained crystals (**4a–c**) were purified by recrystallization from petroleum ether.

## *RESULTS AND DISCUSSION*

### *Synthesis of 3-Methyl-1,2,4-benzotriazines* **2a–f**

The different 2-nitrophenylhydrazones of pyruvic acid **1a–f** shown in Table 1 were obtained according to the method of Schwesinger et al. [4]. At room temperature, the nitrophenylhydrazines reacted readily with sodium pyruvate in the presence of a 35% ethanolic solution of perchloric acid (Eq. (1)). The corresponding phenylhydrazones were instantaneously precipitated as yellow crystals. The yields of the corresponding phenylhydrazones were quantitative and generally higher than 90%. With the exception of **1a**, they all recrystallized from ethanol and their melting points were found to be in the range of 200–230°C. It is interesting that the reaction

**TABLE 1** Characteristic Results of 2-Nitrophenylhydrazones of Pyruvic Acid **1a–f**[a]

| | | | | | | *Elemental Analysis Found (Calculated)* | | |
|---|---|---|---|---|---|---|---|---|
| | $R^1$ | $R^2$ | *Yield (%)* | *mp (°C)* | *Molecular Formula* | *C (%)* | *H (%)* | *N (%)* |
| **1a** | H | H | 95 | 227–228 | $C_9H_9N_3O_4$ (232.2) | 48.39 (48.43 | 4.25 4.06 | 18.76 18.83) |
| **1b** | Cl | H | 92 | 228–229 | $C_9H_8ClN_3O_4$ (257.6) | 42.03 (41.96 | 3.21 3.13 | 16.45 16.31) |
| **1c** | H | Cl | 95 | 225–229 | $C_9H_8ClN_3O_4$ (257.6) | 41.97 (41.96 | 3.17 3.13 | 16.20 16.31) |
| **1d** | Br | H | 95 | 236–237 | $C_9H_8BrN_3O_4$ (302.1) | 35.92 (35.78 | 2.55 2.67 | 14.07 13.91) |
| **1e** | H | Br | 95 | 232–233 | $C_9H_8BrN_3O_4$ (302.1) | 35.59 (35.78 | 2.85 2.67 | 14.08 13.91) |
| **1f** | $NO_2$ | Cl | 90 | 201–202 | $C_9H_7ClN_4O_6$ (268.2) | 35.85 (37.72 | 2.37 2.33 | 18.60 18.52) |

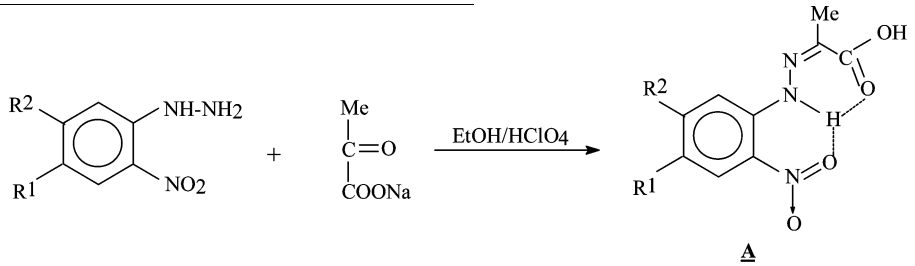
[a]All compounds decomposed beyond their melting points.

**TABLE 2** Spectral Data of 2-Nitrophenylhydrazones of Pyruvic Acid **1a–f**

| | *UV–Vis (EtOH)* $\lambda_{max}$ *(log* $\varepsilon$*)* | *IR* ($\nu$, $cm^{-1}$) *(KBr)* | | *$^1$H NMR* ($\delta$, *ppm) (DMSO-$d_6$)* | |
|---|---|---|---|---|---|
| | | *NH, CO* | *$CH_3$* | *H-Ar* | *NH* |
| **1a** | 223 (4.03)<br>281 (4.10)<br>303 (4.17)<br>416 (3.82) | 3295; 1670 | 2.34 (s, 3H) | 7.24 (t, 1H)<br>7.91 (t, 1H)<br>8.20 (dd, 1H)<br>8.33 (dd, 1H) | 10.79 (s, 1H) |
| **1b** | 207 (4.11)<br>308 (4.20)<br>422 (3.79) | 3320; 1720 | 2.33 (s, 3H) | 7.92 (dd, 1H)<br>8.18 (d, 1H)<br>8.29 (d, 1H) | 10.68 (s, 1H) |
| **1c** | 224 (3.85)<br>287 (4.15)<br>301 (4.13)<br>407 (3.68) | 3305; 1675 | 2.38 (s, 3H) | 7.25 (dd, 1H)<br>8.19 (d, 1H)<br>8.30 (d, 1H) | 10.75 (s, 1H) |
| **1d** | 211 (4.15)<br>307 (4.25)<br>422 (3.84) | 3315; 1710 | 2.31 (s, 3H) | 8.10 (d, 2H)<br>8.43 (d, 1H) | 10.70 (s, 1H) |
| **1e** | 222 (3.73)<br>288 (4.00)<br>300 (3.99)<br>404 (3.86) | 3305; 1690 | 2.37 (s, 3H) | 7.37 (dd, 1H)<br>8.23 (d, 1H)<br>8.40 (d, 1H) | 10.69 (s, 1H) |
| **1f** | 207 (4.17)<br>277 (4.13)<br>346 (4.11) | 3305; 1695 | 2.41 (s, 3H) | 8.47 (s, 1H)<br>9.09 (s, 1H) | 10.83 (s, 1H) |

was diastereoselective, that is, the isomer (*Z*) was exclusively formed as revealed by $^1$H NMR analysis (Table 2); a single downfield peak was observed at $\delta$ 10.68–10.88 ppm, and was assigned to the proton affixed to the nitrogen atom. The exclusive formation of the (*Z*) isomer is probably due to the created hydrogen bonding forming two six-membered heterocyclic moieties as illustrated in **A**. Moreover, thin layer chromatography analysis using different eluting systems revealed one spot, indicating the presence of a single compound. The formation of only the (*Z*) isomer was also observed in a similar work [27]. It should be noted that in neutral or slightly acidic media, the synthesis of the methyl and ethyl pyruvate of nitrophenylhydrazones gave a mixture of (*E*) and (*Z*); whereas, in the presence of an appreciable amount of HCl or $HClO_4$, only the (*E*) isomer was detected. The high melting points of **1a–f** also suggest the (*Z*) conformation as reported [27]. Also, in this line, Zeghough et al. [28] outlined the synthesis of 3,3′-dichloro-4,4′-diphenylene dihydrazone ethyl pyruvate and obtained a yellow crystal which melted at 215–218°C.

Elemental analysis results also supported the success of the reaction providing the expected products. Infrared spectroscopy showed two distinct bands fluctuating between 3295–3320 and 1670–1720 $cm^{-1}$ attributed to the absorptions of NH and C=O of the carboxylic group, respectively. The broad bands at around 3295–3320 $cm^{-1}$, which appeared in the IR spectra, suggest the formation of a hydrogen bonding mentioned above.




**A**

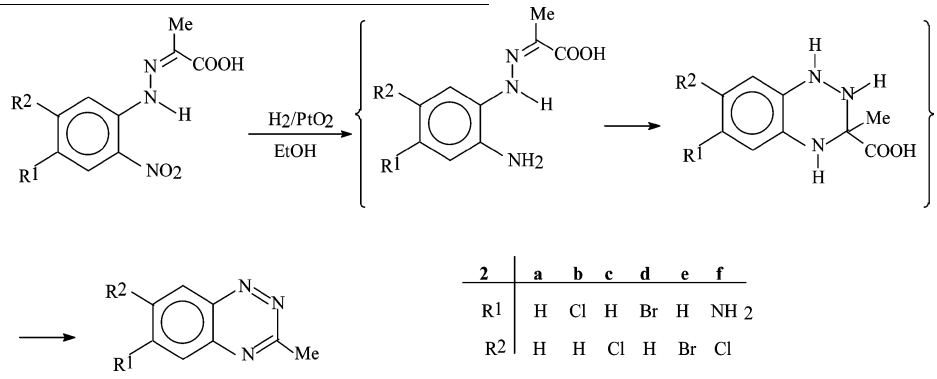
| 1 | a | b | c | d | e | f |
|---|---|---|---|---|---|---|
| R1 | H | Cl | H | Br | H | $NO_2$ |
| R2 | H | H | Cl | H | Br | Cl |

(1)

**TABLE 3** Characteristic Results of 3-Methyl-1,2,4-benzotriazines **2a–f**

| | $R^1$ | $R^2$ | Yield (%) | mp (°C) | Molecular Formula | Elemental Analysis Found (Calculated) C (%) | H (%) | N (%) |
|---|---|---|---|---|---|---|---|---|
| **2a** | H | H | 69 | 94–95 | $C_8H_7N_3$ (145.2) | 65.80 (66.19 | 4.86 4.86 | 28.88 28.95) |
| **2b** | Cl | H | 57 | 112–114 | $C_8H_6ClN_3$ (179.6) | 53.37 (53.50 | 3.41 3.37 | 23.32 23.40) |
| **2c** | H | Cl | 52 | 136–137 | $C_8H_6ClN_3$ (179.6) | 53.72 (53.50 | 3.31 3.37 | 23.18 23.40) |
| **2d** | Br | H | 52 | 117–118 | $C_8H_6BrN_3$ (224.1) | 43.01 (42.88 | 2.68 2.70 | 18.67 18.75) |
| **2e** | H | Br | 53 | 127–128 | $C_8H_6BrN_3$ (224.1) | 42.76 (42.88 | 2.71 2.70 | 18.88 18.75) |
| **2f** | $NO_2$ | Cl | 63 | 233–234 | $C_8H_7ClN_4$ (194.6) | 49.52 (49.37 | 3.58 3.63 | 28.61 28.79) |

Under atmospheric pressure and at room temperature, the hydrogenation of different phenylhydrazones **1a–f** was conducted in ethanol and in the presence of $PtO_2$ as a catalyst (Eq. (2)). The nitro group is first reduced to the amino group followed by a cyclization (in the case of **1f**, the two nitro groups are, obviously, both reduced leading to the corresponding **2f**). The intramolecular cyclization [9] is promoted by the nucleophilic attack of the nitrogen lone electron pair on the adjacent amino group upon the carbon of the C=N bond. This attack is followed by a concomitant decarboxylation and an air oxidation affording the desired products **2a–f**. In Eq. (2),




(2)

parent isomers **2c** and **2e**, and this was not the case for the phenylhydrazones. The elemental analysis results indicate the authenticity of the products. The latter were also characterized by UV–Vis, IR, and $^1$H NMR spectra, and the results are compiled in Table 4. The IR spectra revealed the disappearance of the bands at 3295–3320 and 1670–1720 $cm^{-1}$ of the secondary amine and the carbonyl, respectively. Moreover, the singlet peak at $\delta$ 10.68–10.88 ppm was absent in the $^1$H NMR spectra, suggesting that cyclization had occurred. The $^1$H NMR results shown in Table 4 are in a good agreement with the molecular structures of the products **2a–f**.

the intermediate products are illustrated in order to provide an insight into the occurrence of the final products. These products (**2a–f**) were obtained in moderate yields as can be seen in Table 3. The product **2a** was formed in 69% yield, which is the highest. It is interesting to note that the melting points depend on the nature of the substituents and their positions on the phenyl ring. For example, the melting points of **2b** and **2d** are lower than those of their

It is worth mentioning that attempts were made to use a number of other reducing agents to convert the nitro group of the phenylhydrazones into the amino functionality and either they failed or the yields were not satisfactory. Both systems, such as metals in acidic media (Zn/HCl, Zn/AcOH, Sn/AcOH) and sodium bisulfite (or sodium borohydride), were inefficient. However, sodium dithionite and the Fe/aq. NaCl system were

**TABLE 4** Spectral Data of 3-Methyl-1,2,4-benzotriazines **2a–f**

| | *UV–Vis (EtOH)* $\lambda_{max}$ *(log* $\varepsilon$*)* | *Solvent* | *$^1$H NMR ($\delta$, ppm)* |
|---|---|---|---|
| **2a** | 234 (4.03) | $CDCl_3$ | 3.10 (s, 3H) |
| | 303 (3.38) | | 7.60–8.50 (m, 4H) |
| | 332 (3.20) | | |
| | 446 (2.48) | | |
| **2b** | 237 (4.30) | $CDCl_3$ | 3.06 (s, 3H) |
| | 317 (3.74) | | 7.69 (dd, 1H) |
| | 443 (2.43) | | 7.90 (d, 1H) |
| | | | 8.38 (d, 1H) |
| **2c** | 237 (4.49) | $CDCl_3$ | 3.13 (s, 3H) |
| | 294 (3.41) | | 7.83 (d, 2H) |
| | 345 (3.46) | | 8.40 (d, 1H) |
| | 454 (2.46) | | |
| **2d** | 242 (4.30) | $CDCl_3$ | 3.07 (s, 3H) |
| | 322 (3.83) | | 7.80 (dd, 1H) |
| | 448 (2.47) | | 8.09 (d, 1H) |
| | | | 8.29 (d, 1H) |
| **2e** | 239 (4.44) | $CDCl_3$ | 3.08 (s, 3H) |
| | 292 (3.48) | | 7.76 (d, 1H) |
| | 344 (3.49) | | 7.95 (dd, 1H) |
| | 447 (2.34) | | 8.61 (d, 1H) |
| **2f** | 224 (4.34) | DMSO-$d_6$ | 2.74 (s, 3H) |
| | 270 (4.34) | | 6.83 (s, 1H) |
| | 402 (3.90) | | 7.09 (s, 2H) |
| | | | 8.26 (s, 1H) |

successful but afforded yields of only 30 and 10%, respectively.

### *Synthesis of 3-Phenyl-1,2,4-benzotriazines* **4a–c**

First, the reaction of the corresponding 2-nitrophenylhydrazines with benzoimino-ester afforded the 2-nitrophenylhydrazono-ethers **3a–c** (Eq. (3)). Some other 2-nitrophenylhydrazono-ethers that resulted from the reaction of imino-esters and 2-nitrophenylhydrazines were reported earlier, and served as precursors for the synthesis of the corresponding 1,2,4-benzotriazines [2]. In the present work, the used imino-ester was made using the Pinner reaction starting with benzonitrile. The formation of hydrazono-ethers **3a–c** was not as instantaneous as that of hydrazones **1a–f**, hinting probably at the effect of the two six-membered heterocyclic moieties shown in **A**. The precipitation of **3a–c** indeed occurred only after 1 h of reaction, but the products were obtained in a high purity. Second, these highly pure hydrazono-ethers were hydrogenated at room temperature in the presence of Pt/C as a catalyst (Eq. (4)). The reaction took place via a tetrahydro intermediate, as for 3-methyl-1,2,4-benzotriazines, which

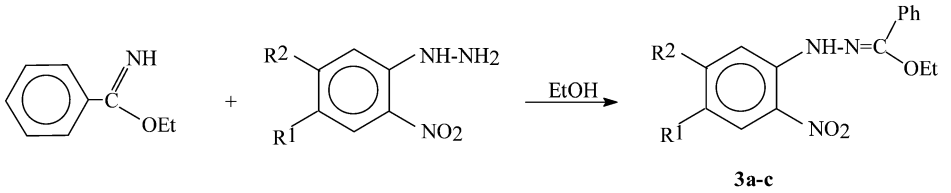
**TABLE 5** Characteristic Results of 3-Phenyl-1,2,4-benzotriazines **4a–c**

| | | | | | | *Elemental Analysis Found (Calculated)* | | |
|---|---|---|---|---|---|---|---|---|
| | *$R^1$* | *$R^2$* | *Yield (%)* | *mp (°C)* | *Molecular Formula* | *C (%)* | *H (%)* | *N (%)* |
| **4a** | H | H | 75 | 126–127 | $C_{13}H_9N_3$ (207.2) | 75.34 (75.12 | 4.38 4.29 | 20.27 20.45) |
| **4b** | H | Cl | 72 | 169–170 | $C_{13}H_8ClN_3$ (241.7) | 64.60 (64.48 | 3.34 3.17 | 17.39 17.25) |
| **4c** | Br | H | 84 | 136–138 | $C_{13}H_8BrN_3$ (286.1) | 54.56 (54.39 | 2.82 2.98 | 14.69 14.51) |

**TABLE 6** Spectral Data of 3-Phenyl-1,2,4-benzotriazines **4a–c**

| | *UV–Vis (EtOH)* $\lambda_{max}$ *(log* $\varepsilon$*)* | *IR ($\nu$, $cm^{-1}$) (KBr)* | *$^1$H NMR ($\delta$, ppm) ($CDCl_3$)* |
|---|---|---|---|
| **4a** | 214 (4.09) | 1500 | 7.42–7.58 (m, 3H) |
| | 263 (4.31) | 1450 | 7.65–8.02 (m, 3H) |
| | 356 (2.47) | 1325 | 8.33–8.50 (m, 1H) |
| | | | 8.60–8.75 (m, 2H) |
| **4b** | 216 (4.13) | 1495 | 7.37–7.62 (m,3H) |
| | 263 (4.36) | 1405 | 7.76 (q, 1H) |
| | 370 (3.59) | 1310 | 7.96 (d, 1H) |
| | 463 (2.45) | | 8.41 (d, 1H) |
| | | | 8.56–8.75 (m, 1H) |
| **4c** | 217 (4.17) | 1495 | 7.37–7.63 (m, 3H) |
| | 270 (4.34) | 1410 | 7.79 (dd, 1H) |
| | 354 (3.14) | 1310 | 8.16 (d, 1H) |
| | 454 (2.45) | | 8.29 (d, 1H) |
| | | | 8.50–8.75 (m, 2H) |

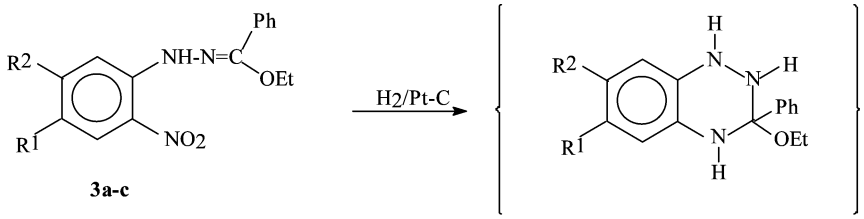
led to the products **4a–c** upon evaporation of ethanol and subsequent oxidation by air. Interestingly, the overall yields of **4a–c** were higher than 72%. Elemental analyses and spectral data shown in Tables 5 and 6, respectively, confirmed the products structures. $^1$H NMR results for all products showed peaks at 7.37–8.75 ppm pertaining to the protons of the two phenyl rings. The UV–Vis spectra revealed bands in the UV region (214–270 nm) and two to three bands in the visible region (354–463 nm). All products decomposed beyond their melting points which are higher than those of their methyl-1,2,4-benzotriazine analogues (**2a, 2c, 2d**).



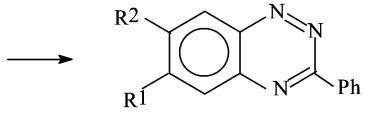

**3a-c**

| 3 | a | b | c |
|---|---|---|---|
| R1 | H | H | Br |
| R2 | H | Cl | H |

(3)




**3a-c**




**4a-c**

| 4 | a | b | c |
|---|---|---|---|
| R1 | H | H | Br |
| R2 | H | Cl | H |

(4)

## *CONCLUSIONS*

3-Methyl and 3-phenyl-1,2,4-benzotriazines were prepared via two-step syntheses. The procedure is facile and the work-up, including the experimental set-up and the reaction conditions, is not strenuous, clean, and overall not time consuming. The yields of the phenylhydrazones and the corresponding starting materials for the 3-methyl-1,2,4-benzotriazines are high. Those of the benzotriazines are acceptable but not as quantitative as for the phenylhydrazones. However, the yields of the 2-nitrophenylhydrazonoethers and their corresponding 3-phenyl-1,2,4-benzotriazines are, however, satisfactory.

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