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Bi(CF₃)₃/Cu(OCOCH₃)₂ — a new system for the synthesis of 2-trifluoromethylcycloalkan-1-ones, trifluoromethylanilines and phenyl(trifluoromethyl)sulfane

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Dedicated to Prof. Dr. Ralf Miethchen on the occasion of his 60th birthday.

Abstract

 $Bi(CF_3)_3$ reacts in the presence of equimolar amounts of $Cu(OCOCH_3)_2$ and 1-morpholinocyclopentene to give 1-morpholino-2-trifluoromethylcyclopentene in 83% yield. This compound as well as intermediately formed 1-morpholino-2-trifluoromethylcyclohexene can easily be converted into the corresponding cycloketones (78 and 41% yield) using the Swarts procedure. In absence of a copper(II) source, no reactions were observed.

The reaction of the system $Bi(CF_3)_3/Cu(OCOCH_3)_2$ with *N*,*N*-diethylaniline gave an 1.7: 1 isomer mixture of 2-trifluoromethyl and 4-trifluormethyl-*N*,*N*-diethylanilines in 48% yield.

With tetrabutylammonium thiophenolate, mixtures of phenyl(trifluoromethyl)sulfane and diphenyldisulfane were obtained. Mechanisms are discussed. © 2000 Elsevier Science S.A. All rights reserved.

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1. Introduction

The use of organobismuth reagents and especially arylbismuth derivatives has been the subject of several reviews [1–3]. There is a great number of publications gathering on organobismuth(V) derivatives in different fields of organic synthesis; however, the use of bismuth(III) compounds, especially alkyl derivatives, is of limited scope to date. Triphenylbismuth in the presence of Cu(II) ions was used as an *O*- and *N*-phenylating reagent in reactions with alcohols, phenols [4–7] and amines [8]. Palladium acetate mediated reactions of Bi(C₆H₅)₃ with acid chlorides in the presence of triethylamine gave the corresponding phenones in good yields [9] whereas reactions with acetyl chloride without any catalyst gave only poor amounts of acetophenone [10]. *N*-phenylation was also observed in reactions of Bi(C₆H₅)₃ and isocyanide complexes of palladium [11]. In a

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comparative study, we found out that $Bi(C_6H_5)_3$ and $Bi(CF_3)_3$, gave benzophenone and α, α, α -trifluoroacetophenone, respectively, in better than 65% yield [12].

In extension of our previous work on reactions of tris(perfluoroorgano)bismuth, we herein report on synthetic applications of $Bi(CF_3)_3$ in $Cu(OCOCH_3)_2$ mediated reactions with different organic substrates.

2. Results and discussion

2.1. The reaction of $Bi(CF_3)_3$ and $Cu(OCOCH_3)_2$

Bi(CF₃)₃ was used as a trifluoromethylating reagent in reactions with AgONO₂ to yield Ag[Ag(CF₃)₄] [13] and in reactions with TeCl₄ to give Te(CF₃)₂Cl₂ as well as in further metallorganic systems [14].

In CH₃CN solution, Bi(CF₃)₃ and Cu(OCOCH₃)₂ react giving Bi(CF₃)₂(OCOCH₃) and Bi(CF₃)(OCOCH₃)₂ as the only species detectable in the ¹⁹F NMR spectra of the reaction mixtures. After a period of 2 days, no resonances of fluoroorganobismuth or copper(I) compounds were detected in the ¹⁹F NMR spectra but CH₃COF, CHF₃,

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 CF_3CH_2CN , C_2F_4 and $c-C_3F_6$ were unambiguously identified. A broadened singlet at -75.8 ppm might be assigned to $CH_3C(O)CF_3$. Further signals of low intensity were detected in the region between -58 and -83 ppm which could not be assigned. CHF_3 , CO and CO₂ absorption bands were detected in the IR spectrum of the gas phase.

The change in colour from deep turquoise to pale brown may be taken as a hint that all copper(II) ions had been converted to lower oxidation state copper species.

The variety of reaction products reveals that copper(II) species, probably $Cu(CF_3)(OCOCH_3)$, had first been formed which decomposed by elimination of CF_3 radicals and difluorocarbene.

 $Bi(CF_3)_3 + Cu(OCOCH_3)_2$

 $\rightarrow Bi(CF_3)_2(OCOCH_3) + [Cu(CF_3)(OCOCH_3)]$

 $[Cu(CF_3)(OCOCH_3)] \rightarrow {}^{\bullet}CF_3 + CuOCOCH_3$

 $[Cu(CF_3)(OCOCH_3)] \rightarrow : CF_2 + [CuF(OCOCH_3)]$

With the present knowledge on the stability of fluoroalkylcopper(III) species, intermediate formation of trifluoromethylcopper(III) compounds can virtually be excluded [15–19].

2.2. The reaction of $Bi(CF_3)_3/Cu(OCOCH_3)_2$ and 1morpholinocycloalkenes

In a previous paper, we described the reactions of Zn(CF₃)Br.₂CH₃CN with 1-morpholinocycloalkenes giving 2-difluoromethyl-1-cyanomethyl derivatives [20]. By contrast, Wakselman and coworkers [21] obtained trifluoromethylcycloalkanones after acidic hydrolysis without isolation of intermediates.

The overall reaction of $Bi(CF_3)_3$, $Cu(OCOCH_3)_2$ and 1morpholinocycloalkenes giving 1-morpholino-2-trifluoromethylcycloalkenes which are converted into the corresponding cyclic ketones using the Swarts procedures may be summarized as follows (Scheme 1).

The progress of the reaction was monitored by recording 19 F NMR spectra either in presence of the organic substrate or not. In both cases, the resonance of Bi(CF₃)₂(OCOCH₃) was identified by the quartet splitting of the 13 C-satellites indicating the magnetic inequivalence of two CF₃ groups bond to the bismuth atom. When the morpholino derivative

was present mainly the first product shown in Scheme 1 was formed; in the absence of the morpholino derivative only CHF₃ was detected. In both reactions, evidence was found for the formation of traces of Bi(CF₃)(OCOCH₃)₂. No reactions were observed mixing Bi(CF₃)₃ and 1-morpholinocycloalkenes. Altogether, these results implicate that hypervalent copper species (hypervalent in the sense of Cu(II) or Cu(III)) are the reactive trifluoromethylating reagents in these systems.

Therefore, the mechanistic approaches formulated for the arylation of alcohols, phenols and amines by triarylbismuth have to be taken into account.

They are based on results of Barton et al. [8] and Dodonov and coworkers [4–7]. Barton et al. studied reactions of $Bi(C_6H_5)_3$, $Cu(OCOCH_3)_2$ and primary amines. They suggested in a primary step the formation of a bismuth(V) compound, namely $Bi(C_6H_5)_3(OCOCH_3)_2$, which reacts in the presence of "freshly" generated copper(I) ions via a copper(III) intermediate to give *N*-phenylated amines; this mechanistic approach was supported by reactions with hypervalent iodine compounds [22].

The final results of the work of Dodonov et al. [5] with alcohols were similar for products and yields.

Our results in estimating the oxidation potential of $Bi(CF_3)_3$ (above 3.0 V in CH₃CN) [14] and our failed attempts to oxidize $Bi(CF_3)_3$ with elemental halogens, XeF_2 and $[XeF][MF_6]$ (M = As, Ta) [23] together with knowledge on the stability of fluoroalkylcopper(III) compounds [15–19] show that these previous mechanistic approaches are not plausible for the reactions investigated by us. Therefore, we propose a different reaction sequence on the basis of radical or possibly radical–ionic decay of trifluoromethylcopper(II) intermediates (Scheme 2).

All reactions and manipulations were carried out in commercially available glassware; therefore, the influence of daylight on the radical decay of intermediately formed $[Cu(CF_3)(OCOCH_3)]$ and $Cu(OCOCH_3)_2$ [24] has also to be taken into account for the formation of radicals and the reduction of Cu(II) ions.

This mechanism explains the formation of 1-morpholino-2-trifluoromethylcycloalkenes.

The second reaction step, the acidic hydrolysis, is the final step of the Swarts reaction to convert enamines into ketones (Scheme 3).



Scheme 1. Reaction sequence for the formation of 1-trifluoromethylcycloalkan-2-ones (n = 1, 2).



Scheme 2. Proposed reaction sequence for the trifluoromethylation of 1-morpholinocycloalkenes with the system Bi(CF₃)₃/Cu(OCOCH₃)₂.



Scheme 3. The acidic hydrolysis of 1-morpholinocycloalkenes (Swarts procedure).

2.3. The reaction of $Bi(CF_3)_3/Cu(OCOCH_3)_2$ and N,Ndiethylaniline

Bi(CF₃)₃ reacts with *N*,*N*-diethylaniline in the presence of Cu(OCOCH₃)₂ to give a mixture of the 2-CF₃-, 4-CF₃-*N*,*N*-diethylaniline and CHF₃ in a ratio of 1.7:1:2.7. The ratio was determined by integrating the ¹⁹F NMR signals of the mixture in a gas-tight NMR tube. By contrast, the light-induced reaction of Te(CF₃)₂ and the same substrate gave a 1:1:1:3 mixture of all three isomers and CHF₃ [25].

Changes of the colours of the reaction mixture of $Bi(CF_3)_3/Cu(OCOCH_3)_2/N$,*N*-diethylaniline appear to be noteworthy. The colour changed from bright turquoise via dark blue or deep violet to become finally nearly colourless. During the reaction period solids of white, red and grey colour precipitated irrespective of stoichiometry and conditions. Qualitative analysis exhibited the precipitate consisted mainly of copper, bismuth, fluoride and acetate. However, no explanation for this reaction and the phenomena observed can be given.

2.4. The reaction of $Bi(CF_3)_3/Cu(OCOCH_3)_2$ and $[(n-C_4H_9)_4N][SC_6H_5]$

Several different pathways are known for the synthesis of $C_6H_5SCF_3$ [26–28]. The reaction of the system investigated is a new approach to $C_6H_5SCF_3$. The room temperature reaction of Bi(CF₃)₃/Cu(OCOCH₃)₂ and [(*n*-C₄H₉)₄N] [SC₆H₅] gave a product mixture of C₆H₅SCF₃ and



The by-products formed were CH₃COF, c-C₃F₆ and Bi(CF₃)(OCOCH₃)₂ as well as derivatives showing ¹⁹F resonances between -65 and -76 ppm. Also a low-intensity signal of the starting material, Bi(CF₃)₃, was detected. Variation of stoichiometry and reaction conditions did not lead to higher selectivity. From one reaction, an isomer mixture of trifluoromethylanilines was isolated by GC in 48% yield (2-CF₃:4-CF₃ = 1.7 : 1).

 $(C_6H_5S)_2$. On the basis of experimental results, two parallel reactions must be formulated: radical trifluoromethylation of the thiophenolate ion

$$\begin{split} & C_6H_5S^- + {}^{\bullet}CF_3 \rightarrow [C_6H_5SCF_3]^{\bullet-} \\ & [C_6H_5SCF_3]^{\bullet-} + Cu(OCOCH_3)_2 \\ & \rightarrow C_6H_5SCF_3 + CuOCOCH_3 + CH_3COO \end{split}$$

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and oxidative coupling of two thiophenolate anions mediated by copper(II) ions [29].

$$\begin{split} & 2C_6H_5S^- + 2Cu(OCOCH_3)_2 \\ & \rightarrow (C_6H_5S)_2 + 2CuOCOCH_3 + 2CH_3COO \end{split}$$

Variation of reaction parameters did not give evidence for the selective formation of $C_6H_5SCF_3$.

3. Experimental

All reactions were carried out in a dry nitrogen atmosphere using Schlenk techniques. The work-up procedures did not require this technique.

N,*N*-Diethylaniline was purchased from Aldrich, Cu(O-COCH₃)₂·H₂O from Fluka and used after careful dehydration in vacuo. The following products were synthesized according to literature procedures: 1-morpholinocyclopentene and hexene [30], $[(n-C_4H_9)_4N][SC_6H_5]$ by analogy to [31], Bi(CF₃)₃ [13,32]. All solvents were purified according to literature procedures [33].

¹⁹F NMR spectra were recorded on Bruker spectrometers WP 200 or AC 200 operating at 188.3 MHz, ¹H NMR spectra on a Gemini 200 spectrometer. Positive shifts denote resonances occuring downfield from the external standard CCl_3F (¹⁹F) and the internal standard [(CH₃)₃Si]₂O (¹H).

3.1. The system Bi(CF₃)₃/Cu(OCOCH₃)₂/CH₃CN

Bi(CF₃)₃ (0.14 g (0.33 mmol)) was added to a solution of 0.12 g (0.66 mmol) carefully dried Cu(OCOCH₃)₂ in 2 ml CH₃CN. The turquoise solution was stirred at ambient temperature for several days. The course of the reaction was monitored by recording ¹⁹F NMR spectra. After a reaction time of 5 h, the spectrum was dominated by the resonance of the starting material $Bi(CF_3)_3$ (-33.7 ppm). Low-intensity resonances for Bi(CF₃)₂(OCOCH₃) (-36.1 ppm) and $Bi(CF_3)(OCOCH_3)_2$ (-41.0 ppm) were also detected. ¹⁹F NMR spectra recorded after 2 days, exhibited the signals of CH_3COF (+50.0 ppm, q, ${}^{3}J({}^{19}F$ -¹H) = 7.0 Hz), CHF₃ (-78.9 ppm, d, ${}^{2}J({}^{19}F_{-}^{-1}H) =$ 79.3 Hz), CF₃CH₂CN (-64.3 ppm, t, ${}^{3}J({}^{19}F_{-}^{-1}H) =$ 9.0 Hz), C₂F₄ (-133.0 ppm, s) and c-C₃F₆ (-156.5 ppm, s). One broad signal at -75.8 ppm, probably CH₃C(O)CF₃ or the corresponding ketal, as well as several low-intensity signals covering the region between -58 and -83 ppm could not be assigned. In the course of the reaction, the colour of the reaction mixture changed from bright turquoise to pale brown; a red-brown solid precipitated. The gas-phase IR-spectrum (7 hPa, KBr-plates) showed only the absorption bands of CHF₃, CO₂ and CO.

3.2. The system $Bi(CF_3)_3/Cu(OCOCH_3)_2/1$ morpholinocyclopentene

To a solution of 0.70 g (1.68 mmol) $Bi(CF_3)_3$ in 5 ml $CH_3CN,\ 0.30$ g (1.65 mmol) $Cu(OCOCH_3)_2$ and 0.15 g

(0.98 mmol) 1-morpholinocyclopentene were added at 0° C. The reaction mixture was stirred for 1 h at 0° C and an additional hour at ambient temperature. The residue was filtered off, and all volatile compounds were removed by vacuum distillation at room temperature. The remaining product was extracted with low-boiling petroleumbenzene. After evaporation of the eluent, 1-morpholino-2-trifluoro-methylcyclopentene remained as an analytically pure pale yellow oil in 83% yield (0.18 g).

Elemental analysis [found (calculated)]: C 54.01 (54.29), H 6.12 (6.38), N 6.25 (6.33), F 25.54 (25.76)%.

NMR data (CDCl₃): ¹⁹F: -69.2 ppm, s; ¹H: 3.55 ppm, m, 4H; 2.74 ppm, m, 4H; 1.82–2.24 ppm, m, broad, 6H.

After hydrolysis with cold aqueous hydrochloric acid (20%) and extraction with diethylether followed by evaporation of the ether, 2-trifluoromethylcyclopentanone was obtained in 78% yield (bp 74–78°C at 25 Torr).

Elemental analysis [found (calculated)]: C 47.25 (47.38), H 4.80 (4.64)%.

The NMR spectra agreed well with published data [34].

3.3. The system $Bi(CF_3)_3/Cu(OCOCH_3)_2/1$ morpholinocyclohexene

The reaction was carried out by analogy to the method described above. The complete reaction mixture was hydrolyzed with cold aqueous hydrochloric acid (20%). After extraction with diethyl ether and evaporation of the solvent 2-trifluoromethylcyclohexanone was obtained as an oily liquid in 41% yield with a boiling point of 94–96°C (33 Torr) (cf. literature data $80-82^{\circ}$ C (18 Torr) [21] and $90-92^{\circ}$ C (30 Torr) [35]). The NMR data were identical with those given in [21,35].

3.4. The system $Bi(CF_3)_3/Cu(OCOCH_3)_2/N,N-diethylaniline$

A mixture of 0.22 g (0.53 mmol) Bi(CF₃)₃, 0.10 g (0.55 mmol) Cu(OCOCH₃)₂ and 0.04 g (0.27 mmol) *N*,*N*-diethylaniline in 5 ml CH₃CN was stirred for 72 h at ambient temperature. ¹⁹F NMR spectra were recorded to determine the composition of the reaction mixture. Integrating the signals of CHF₃ and 2- as well as 4-trifluoromethylaniline gave a ratio of approximately 1:1. The integrals of the two trifluoromethylaniline isomers exhibited a ratio of 1.7:1 favouring the 2-isomer. The ¹⁹F NMR data agreed well with those given in [25]. Further signals were detected for CH₃COF, Bi(CF₃)₃, Bi(CF₃)(OCOCH₃)₂, c-C₃F₆; low-intensity signals were covering the region between -65 and -76 ppm.

Variations of molar ratios and reaction times did not lead to an increase in selectivity. During all reactions, marked changes in colour were observed. In all cases, the originally bright turquoise colour changed to dark blue or deep violet. Irrespective of stoichiometry, white, intensive red or metallic grey solids precipitated containing copper, bismuth, fluoride and acetate as determined by qualitative analysis. With extension of the reaction time, the mixtures became vermilion, then colourless. However, in most cases the content of trifluoromethylanilines did not exceed 1/5 of all fluorine containing compounds (integration of the ¹⁹F NMR spectra). Using a 10-fold entry, separation of trifluoromethylanilines was successful via GC. 2-Trifluoromethyl-and 4-trifluoromethyl-*N*,*N*-diethylaminobenzene were isolated in 48% yield (2-CF₃:4-CF₃ =1.67:1).

3.5. The system $Bi(CF_3)_3/Cu(OCOCH_3)_2/$ tetrabutylammonium thiophenolate

A mixture of 0.66 g (1.59 mmol) $Bi(CF_3)_3$, 0.30 g (1.65 mmol) $Cu(OCOCH_3)_2$ and 0.30 g (0.85 mmol) [$(n-C_4H_9)_4N$][SC₆H₅] in 5 ml CH₃CN was stirred for 4 h at ambient temperature. The mixture was extracted with diethylether and finally washed with dilute aqueous NaOH and water. The organic layer was dried with MgSO₄. After evaporating the solvent and the extracting reagent, C₆H₅SCF₃ was distilled in vacuo. C₆H₅SCF₃ was identified by ¹⁹F (-43.1 ppm) [27,28] and ¹H NMR spectroscopy as well as its boiling point ((glass-capillary) 141–142°C) [36]. Significant amounts of (C₆H₅S)₂ were formed as the by-product.

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