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Silver compounds in synthetic chemistry Part 5: Selective syntheses of trifluoromethylketones, $RCOCF_3$, from trifluoromethylsilver, $AgCF_3$, and corresponding acyl chlorides, RCOCl ☆

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Abstract

Trifluoromethylketones of aromatics, heteroaromatics and olefins are formed selectively from reactions of trifluoromethylsilver and the corresponding carboxylic acid chlorides in moderate to excellent yields. The conditions chosen are dependent on the nature of the acyl chloride. Attempts to prepare alkyl(trifluoromethyl)ketones yielded product mixtures of the corresponding acyl fluorides, trifluoromethyl-, pentafluoroethyl- and *n*-heptafluoropropyl ketones.

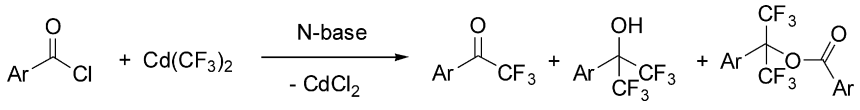
Keywords: Trifluoromethylketones; Silver; Synthesis

1. Introduction

Trifluoromethylketones are valuable building blocks for the synthesis of a variety of compounds which are of interest to synthetic, physical and medicinal chemists due to their important biological and physical properties [1,2].

However, only a few reagents for trifluoroacetyl arenes synthesis are known, for example, the trifluoroacetyl chloride/$AlCl_3$ system [3], trifluoroacetic acid anhydride [4,5] and trifluoroacetyl triflate [6]. Trifluoroacetic acid anhydride and the latter mixed anhydride are applicable only to the trifluoroacylation of activated arenes such as azulene or anthracene, while trifluoroacetyl chloride is difficult to handle due to its low boiling point of −19 °C [7]. An alternative pathway is the reactions of 2-(trifluoroacetoxy)pyridine in the presence of aluminium chloride with benzene, alkylbenzenes, naphthalene and dibenzofuran to give the corresponding trifluoromethylaryl ketones [8]. In most synthetic approaches, $ArCOCF_3$ has been prepared by the reactions of appropriate Grignard reagents with trifluoroacetic acid [9,10] or lithium or ethyl trifluoroacetate [11,12].

Starting with acyl chlorides, in the presence of strong nitrogen bases, perfluoroalkyl ketones were prepared with cadmium reagents $Cd(R_f)_2$ ($R_f = CF_3$, C_2F_5, *i*-C_3F_7, *n*-C_3F_7, *n*-C_4F_9) [13], unfortunately together with a number of by-products (Eq. (1)).



(1)

In the case of reactions with Me_3SiCF_3, acyl chlorides, anhydrides or activated esters can be used to prepare the corresponding bis(trifluoromethyl)carbinols via the intermediate trifluoromethyl ketone [14]. However, these reactions

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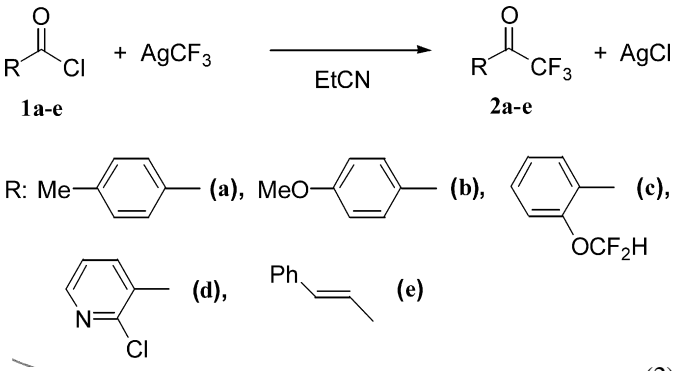
cannot selectively be terminated at the ketone stage [15].

In a recent report, we published a convenient and absolutely selective method for the preparation of 2,3,4,5,6-pentafluorophenones starting from carboxylic acid chlorides and pentafluorophenyl silver under moderate conditions [16]. A selective route to prepare trifluoromethyl silver involves the reaction of trimethyl(trifluoromethyl)silane with silver(I) fluoride in an appropriate solvent such as acetonitrile, propionitrile or DMF at room temperature [17]. In continuation of our earlier work, we present scope and limitations of the trifluoromethylation of acyl chlorides with $AgCF_3$.

2. Results and discussion

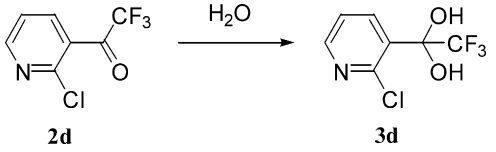
The reactions of the acyl chlorides **1a–e** with trifluoromethylsilver generated in situ proceed selectively in EtCN in the temperature range between −30 °C and ambient temperature giving the corresponding trifluoromethylketones **2a–e** in good yields (Method A) (Eq. (2)).



(2)

Neither the difluoromethoxy group in **2c** nor the chloro ligand in **2d** is affected by this procedure. Under these conditions, *E*-cinnamoyl chloride **1e**, as an example of compounds with a carbonyl function attached to an olefinic double bond, is also converted into the corresponding ketone **2e**.

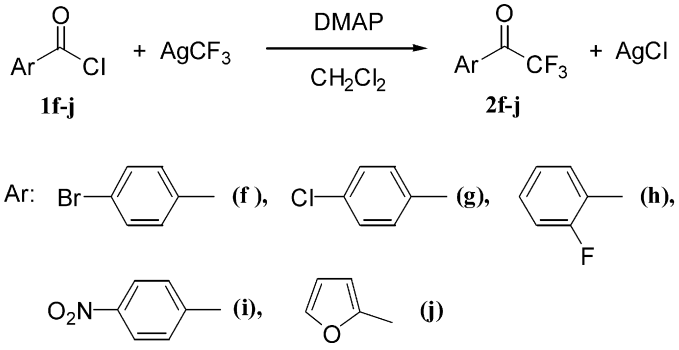
Compound **2d** is very sensitive to moisture and especially in the aqueous working up procedure it is completely converted into the geminal diol **3d** (Eq. (3)).



(3)

Our attempts to obtain ketones with halogen atoms or a nitro group bond to the phenyl group remained unsuccessful by this method. The corresponding acyl fluorides were obtained as the major product in all these cases. Therefore, we applied the conditions found for selective reactions of bis(perfluoroalkyl)cadmium complexes with arylcarbonyl halides in the presence of strong bases such as 4-(*N*,*N*-dimethylamino)pyridine (DMAP) [13] to the $AgCF_3$/RCOCl system. The more or less selective formation of the corresponding perfluoroalkyl ketones may be attributed to the intermediate formation of 1-arylcarbonyl-4-(*N*,*N*-dimethylamino)pyridinium chloride. The formation of the 1-arylcarbonyl-4-(*N*,*N*-dimethylamino)pyridinium chloride has been proved for the 4-NO_2-derivative in CH_2Cl_2 solution. ^{13}C NMR data were in good agreement with those reported for 1-benzoyl-4-(*N*,*N*-dimethylamino)pyridinium chloride in $CDCl_3$ [18]. The +M-effect of the dimethylamino group in the 4-position of the pyridine ring, to suppress the carbonyl activity preventing from further side reactions has been discussed elsewhere [13].

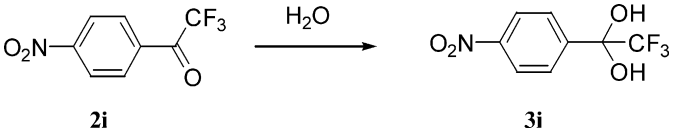
We found that the intermediate formation of 1-carboxyl-4-(*N*,*N*-dimethylamino)pyridinium chlorides in the reactions with trifluoromethylsilver especially with **1f–j** favors the formation of the trifluoromethyl ketones **2f–j** (Method B) (Eq. (4)) which could not be obtained via the procedure mentioned above.



(4)

There was no spectroscopic evidence for substitution of the ring halogens in the reactions with **1f** and **1g**.

Also 4-nitrophenyl(trifluoromethyl)ketone **2i** is very sensitive to moisture or traces of water and is easily converted into the corresponding geminal diol **3i** (Eq. (5)) [13].



(5)

The geminal diols (**3d**, **3i**) can be easily converted into the corresponding ketones (**2d**, **2i**) upon treatment with dicyclohexylcarbodiimide in dichloromethane for 6–8 h at ambient temperature but even short contact times with moisture or using glassware which has not been carefully dried led to signals of the diols in the ^{19}F NMR spectra. The crystal structure of **3i** was solved in the triclinic space group $P\bar{1}$ and not as reported erroneously in "monoclinic, $P1$" [19]; however, crystal and geometry parameters agree excellently with those reported [19].

The main advantage of both reaction pathways is that in neither case evidence is found for the formation of silver salts of the corresponding carbinols nor for substitution of bromine or chlorine atoms bond to the aromatic ring. The only by-products formed are small amounts of trifluoromethane or the corresponding acyl fluorides. Only the reaction of 4-nitrobenzoyl chloride with trifluoromethylsilver in EtCN proceeds with

formation of minor amounts of 1,1,1,3,3,3-hexafluoro-2-(4-nitrophenyl)propanol and the corresponding ester of 4-nitrobenzoic acid similarly as described in Ref. [13].

The attempted conversion of aliphatic acyl chlorides such as nonanoyl chloride and 10-undecenoyl chloride to trifluoromethyl ketones remained unsuccessful or proceeded with very low selectivity. In the presence of DMAP, no reaction occurs at the carbonyl function. However, ^{19}F NMR signals in the regions of the $AgCF_3$ units are shifted, giving rise to the assumption that species such as $AgCF_3$, $[Ag(CF_3)_2]^-$, $[Ag(CF_3)Cl]^-$ and $[AgCl_2]^-$ exchange with each other [20]. In reactions performed in EtCN, the corresponding acyl fluorides are formed as the major products (yields about 50%). A mixture of the corresponding trifluoromethyl-, pentafluoroethyl- and *n*-heptafluoropropyl ketones was isolated from the reaction mixture after removal of the acyl fluoride. An explanation for these findings might be the involvement of difluorocarbene or some carbenoid species.

The reactions of carboxylic acid chlorides and trifluoromethyl silver offer, due to the convenient accessibility of $AgCF_3$ [17], an efficient route for the selective synthesis of a series of trifluoromethylketones as demonstrated for selected examples.

3. Experimental

Schlenk techniques were used throughout all manipulations. Purifications were carried out in ambient atmosphere. NMR spectra of compounds isolated were recorded on a Bruker Avance II 300 (^{1}H, 300.1 MHz; ^{19}F, 282.4 MHz; ^{13}C, 75.4 MHz) spectrometer in $CDCl_3$ solutions unless quoted. External standards were used in all cases (^{1}H, ^{13}C: Me_4Si; ^{19}F: CCl_3F). Chemical shifts (δ) are given in ppm; couplings (J) in Hz. Assignment of all resonances was made using 1D and 2D NMR experiments. Acetone-d_6 was used as an external lock (5 mm tube) in reaction control measurements while an original sample of the reaction mixture was measured in a 4 mm insert; these spectra were run on a Bruker AC 200 spectrometer. Visible melting points were determined using a Stuart melting point apparatus SMP10. C, H and N analyses were carried out with a HEKAtech Euro EA 3000 apparatus. Acyl chlorides were prepared according to standard methods [21] from the corresponding acids, either by treatment with thionyl chloride or oxalyl chloride. All compounds are characterized by melting or boiling points, elemental analyses and NMR spectroscopic data. For known compounds, only melting or boiling points as well as ^{1}H and ^{19}F NMR data are listed, while values of elemental analyses are omitted.

3.1. General procedure (Method A)

To a well-stirred mixture of AgF (0.51 g, 4 mmol) in propionitrile (5 ml) Me_3SiCF_3 (0.71 g, 5 mmol) was added at room temperature. After stirring for 2 h, the formation of $AgCF_3$ was complete. The reaction mixture was cooled to −30 °C and the corresponding acyl chloride (4 mmol) in propionitrile (5 ml) was added drop-wise over a period of 20 min. The well-stirred reaction mixture was warmed slowly to ambient temperature over a period of 3 h, and stirring was prolonged for additional 12 h at ambient temperature. The reaction was terminated after signals of $AgCF_3$ were no longer detected in the ^{19}F NMR spectra. The complete mixture was filtered from the silver salts; diethyl ether (15 ml) was added to the filtrate. The filtrate was washed with a 5% aqueous Na_2CO_3 solution and water. Finally, the organic layer was dried over $MgSO_4$. Ether was evaporated and the residue was distilled under reduced pressure or crystallized.

3.1.1. α,α,α-Trifluoro-4-methylacetophenone (**2a**)

Colourless liquid. Yield 0.58 g (77%), bp 38–39 °C (0.4 mmHg ≅ 5.3×10^{-1} mbar), lit. bp 76–77 °C (18 mmHg) [8]. ^{19}F NMR, δ: −71.8 (s, CF_3); ^{1}H NMR, δ: 8.0 (“d”, 2H, H-2,6, J_{HH} = 8.0), 7.3 (“d”, 2H, H-3,5, J_{HH} = 8.0), 2.5 (s, 3H, CH_3), cf. Ref. [13].

3.1.2. α,α,α–Trifluoro-4-methoxyacetophenone (**2b**)

Colourless liquid. Yield 0.60 g (73%), bp 84–85 °C (0.4 mmHg ≅ 5.3×10^{-1} mbar), lit. bp 122 °C (47 mmHg) [8]. ^{19}F NMR, δ: −71.5 (s, CF_3); ^{1}H NMR, δ: 8.1 (d, 2H, H-2,6, J_{HH} = 8.0), 7.0 (d, 2H, H-3,5, J_{HH} = 8.0), 3.9 (s, 3H, OCH_3), cf. Ref. [13].

3.1.3. α,α,α–Trifluoro-2-difluoromethoxyacetophenone (**2c**)

Colourless liquid. Yield 0.61 g (63%), bp 61–63 °C (0.08 mmHg ≅ 1.0×10^{-1} mbar). ^{19}F NMR, δ: −71.5 (s, 3F, CF_3), −82.5 (d, 2F, $OCHF_2$, $^2J_{FH}$ = 72.6); ^{1}H NMR, δ: 7.3–7.8 (m, 4H, phenyl), 6.57 (t, 1H, ($OCHF_2$), J_{HF} = 72.6). Anal. calcd. for $C_9H_5F_5O_2$: C 45.0, H 2.0. Found: C 44.9, H 1.9.

3.1.4. 2-Chloro-3-trifluoroacetyl-pyridine (**2d**)

Due to the extreme sensitivity of **2d** to moisture only ^{19}F and ^{1}H NMR data were obtained even after treatment of the corresponding diol **3d** with dicyclohexylcarbodiimide.

^{19}F NMR, δ: −73.4 (s, CF_3; $^1J_{F,C}$ = 292, $^2J_{F,C}$ = 36); ^{1}H NMR, δ: 8.47 (dd, 1H, H-6), 7.66 (dd, 1H, H-4), 7.31 (dd, 1H, H-5).

3.1.5. E-1,1,1-Trifluoro-4-phenyl-3-buten-2-one (**2e**)

Colourless liquid. Yield 0.4 g (50%), bp 43–44 °C (0.08 mmHg ≅ 1.0×10^{-1} mbar) [22]. ^{19}F NMR, δ: −78.1 (s, CF_3); ^{1}H NMR, δ 7.96 (d, 1H, = CH, $^3J_{H,H}$ = 16), 7.63 (m, 2H, phenyl), 7.50 (m, 3H, phenyl), 7.02 (d, 1H, =CH, $^3J_{H,H}$ = 16), cf. Ref. [23].

3.2. General procedure (Method B)

To a well-stirred mixture of AgF (0.51 g, 4 mmol) in propionitrile (5 ml) Me_3SiCF_3 (0.71 g, 5 mmol) was added at room temperature. Stirring was maintained for 2 h until the formation of $AgCF_3$ was complete. Propionitrile was evaporated under reduced pressure at ambient temperature and dichloromethane (5 ml) were added. The reaction mixture was cooled to −30 °C and the mixture of the corresponding acyl chloride (4 mmol) and DMAP (4 mmol) in 20 ml of dichloromethane was added during 2 min. The reaction mixture

was gently warmed to ambient temperature and was stirred at ambient temperature for 48 h (compound **2i** for 72 h). The reaction was terminated after signals of $AgCF_3$ were no longer detected in the ^{19}F NMR spectra. The complete mixture was filtered from the silver salts. The filtrate was washed with a 5% aqueous Na_2CO_3 solution and water. Finally, the organic layer was dried over $MgSO_4$. Dichloromethane was distilled off and the remaining residue was distilled under reduced pressure.

Compound **2i** was isolated as follows. The reaction mixture was filtered from the precipitate and dichloromethane was evaporated under reduced pressure at ambient temperature. The residue was washed with anhydrous benzene, filtered and dry HCl gas was bubbled into the filtrate. The precipitate of 4-*N*,*N*-dimethylaminopyridine hydrochloride was filtered off and all volatiles were removed under reduced pressure. **2i** was crystallized from carefully dried *n*-hexane.

3.2.1. α,α,α–Trifluoro-4-bromoacetophenone (2f)

Colourless liquid. Yield 0.62 g (61%), bp 45–47 °C (5×10^{-2} mbar), lit. bp 95 °C (4 mmHg) [23]. ^{19}F NMR, δ: −71.6 (s, CF_3, $^1J_{F,C} = 291$, $^2J_{F,C} = 36$); ^1H NMR, δ: 7.93 (m, 2H, H-2,6), 7.7 (m, 2H, H-3,5); ^{13}C NMR, δ: 179.7 (q, CO, $^2J_{F,C} = 36$), 132.6 (s, C-3,5), 131.4 (s, C-2,6), 131.0 (s, C-4), 128.6 (s, C-1), 116.4 (q, CF_3, $^1J_{F,C} = 291$).

3.2.2. α,α,α,–Trifluoro-4-chloroacetophenone (2g)

Colourless liquid. Yield 0.65 g (78%), bp 63–64 °C (8.7×10^{-2} mbar), lit. bp 84 °C (23 mmHg) [24]. ^{19}F NMR, δ: −71.6 (s, CF_3, $^1J_{F,C} = 291$, $^2J_{F,C} = 36$); ^1H NMR, δ: 8.02 (m, 2H, H-2,6), 7.53 (m, 2H, H-3,5); ^{13}C NMR, δ: 179.5 (q, CO, $^2J_{F,C} = 36$), 142.5 (s, C-4), 131.3 (s, C-2,6), 129.6 (s, C-3,5), 128.2 (s, C-1), 116.4 (q, CF_3, $^1J_{F,C} = 291$).

3.2.3. α,α,α–Trifluoro-2-fluoroacetophenone (2h)

Colourless liquid. Yield 0.55 g (71%), bp 40–42 °C (9.2×10^{-2} mbar), lit. bp 60–62 °C (22 mmHg) [25]. ^{19}F NMR, δ: −74.8 (d, 3F, CF_3, $^5J_{F,F} = 16$, $^1J_{F,C} = 289$, $^2J_{F,C} = 38$), −107.7 (m, 1 F, F-2); ^1H NMR, δ: 7.91 (m, 1H, H-4), 7.70 (m, 1H, H-6), 7.33 (m, 1 H, H-5), 7.25 (m, 1H, H-3); ^{13}C NMR, δ: 179.2 (q, CO, $^2J_{F,C} = 37$), 162.0 (d, C-2, $^1J_{F,C} = 263$), 137.2 (d, C-6, $^3J_{F,C} = 9$), 131.6 (s, C-4), 124.7 (d, C-5, $^4J_{F,C} = 4$), 119.6 (d, C-1, $^2J_{F,C} = 11$), 117.4 (d, C-3, $^2J_{F,C} = 22$), 116.0 (q, CF_3, $^1J_{F,C} = 290$), cf. Ref. [25].

3.2.4. α,α,α–Trifluoro-4-nitroacetophenone (2i)

Yellow crystals. Yield 0.44 g (50%), mp 44–6 °C (*n*-hexane), lit. mp 47 °C [13]. ^{19}F NMR, δ: −71.9 (s, CF_3, $^1J_{F,C} = 290$, $^2J_{F,C} = 37$); ^1H NMR, δ: 8.46 (m, 2H, H-3,5), 8.29 (m, 2H, H-2,6), cf. Ref. [13].

3.2.5. 2-Trifluoroacetylfuran (2j)

Colourless liquid. Yield 0.42 g (61%), bp 24 °C (2.8×10^{-2} mbar), lit. bp 129–131 °C [26]. ^{19}F NMR, δ: −73.7 (s, CF_3, $^1J_{F,C} = 289$, $^2J_{F,C} = 38$); ^1H NMR, δ: 7.84 (m, 1H, H-5), 7.34 (m, 1H, H-3), 6.71 (''dd'', 1H, H-4). ^{13}C NMR, δ: 168.5 (q, CO, $^2J_{F,C} = 37$), 150.2 (s, C-5), 146.8 (s, C-2), 124.2 (s, C-3), 116.1 (q, CF_3, $^1J_{F,C} = 291$), 113.2 (s, C-4).

3.3. Syntheses of 2,2,2-trifluoroethane-1,1-diols (3)

While compound **3d** is formed directly upon exposure of a chloroform solution of **2d** to ambient atmosphere, **3i** was prepared as follows. The mixture of 0.22 g (1.0 mmol) of 4-nitrophenyl(trifluoromethyl)ketone (**2i**), acetone (5 ml) and water (1 ml) was stirred for 30 min at ambient temperature. The solvent was evaporated at reduced pressure and the remaining residue was crystallized from cyclohexane.

3.3.1. 1-(2-Chloro-pyridin-3-yl)-2,2,2-trifluoroethane-1,1-diol (3d)

Colourless crystals. Yield 0.70 g (77%), mp 147–149 °C (glyme). ^{19}F NMR ($(CD_3)_2CO$), δ: −83.4 (s, CF_3, $^1J_{F,C} = 289$, $^2J_{F,C} = 33$); ^1H NMR, δ: 8.45 (dd, 1H, H-6), 8.34 (dd, 1H, H-4), 7.48 (dd, 1H, H-5), 7.01 (s, 2H, OH, $\Delta_{1/2} \approx 4$); ^{13}C NMR, δ: 150.2 (s, 1C, C-6), 149.6 (s, C-2), 140.1 (s, 1C, C-4), 131.7 (s, 1C, C-3), 122.9 (q, CF_3, $^1J_{F,C} = 289$), 122.1 (s, 1C, C-5), 92.1 (q, $C(OH)_2$, $^2J_{F,C} = 33$). Anal. calcd. for $C_7H_5ClF_3NO_2$: C 36.9; H 2.2; N 6.2. Found: C 37.2; H 2.2; N 6.3.

3.3.2. 2,2,2-Trifluoro-1-(4-nitro-phenyl)-ethane-1,1-diol (3i)

Yield 0.22 g (93%), mp 79–80 °C. Lit. mp 79–80 °C [13]. ^{19}F NMR ($(CD_3)_2CO$), δ: −84.5 (s, CF_3, $^1J_{F,C} = 288$, $^2J_{F,C} = 32$); ^1H NMR, δ: 8.31 (m, 2H, H-3,5), 8.02 (m, 2H, H-2,6), 7.03 (m, 2H, OH); ^{13}C NMR, δ: 148.7 (s, 1C, C-4), 144.9 (s, C-1), 128.9 (s, 2C, C-2,6), 123.1 (q, CF_3, $^1J_{F,C} = 288$), 122.9 (s, 2C, C-3,5), 92.9 (q, $C(OH)_2$, $^2J_{F,C} = 32$). Anal. calcd. for $C_8H_6F_3NO_4$: C 40.5; H 2.5; N 5.9. Found: C 40.2; H 2.6; N 5.9; cf. Ref. [19].

X-ray crystal structure analysis of **3i**; cf. Ref. [19]: $C_8H_6F_3NO_4$ (FW = 237.135), triclinic, $P\bar{1}$, $a = 8.734(2)$ Å, $b = 9.684(2)$ Å, $c = 11.345(2)$ Å, $\alpha = 91.82(2)^\circ$, $\beta = 103.60(2)^\circ$, $\gamma = 96.97(2)^\circ$, $V = 923.9(3)$ Å^3, $Z = 4$, $D_{calc.} = 1.705$ g cm^{-3}, $T = 293(2)$ K. X-ray intensities were measured on a IPDS I diffractometer (Stoe & Cie) with graphite-monochromated Mo Kα radiation. The final *R* factor was 0.0469 (Rw = 0.1293 for all data) for 4120 reflections with $I > 2\sigma(I)$. Crystallographic data for the structure have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC-642163. Copies of the data can be obtained, free of charge, on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: +44 1223 336033 or e-mail: deposit@ccdc.cam.ac.uk).

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