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Silver compounds in synthetic chemistry Part 2: A convenient synthesis of 2,3,4,5,6-pentafluorophenones, C_6F_5COR, from pentafluorophenylsilver, AgC_6F_5, and the corresponding acid chlorides, RCOCl☆

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Abstract

2,3,4,5,6-Pentafluorophenones are formed selectively from the reactions of pentafluorophenylsilver and carboxylic acid chlorides in moderate to excellent yields.

Keywords: Pentafluorophenones; Silver; Synthesis

1. Introduction

In one of the first papers dealing with pentafluorophenylsilver, the authors pointed out that AgC_6F_5 is a reagent of particular value in halide substitutions [1]. While the oxidative properties of this reagent have been demonstrated in a variety of reactions [2,3], examples of nucleophilic C_6F_5 group transfers are limited more or less to inorganic compounds [4,5].

Ketones are generally not obtained when acid chlorides are treated with Grignard reagents or lithium compounds [6]. The ketone initially formed directly reacts with a second molecule of these reagents to give the salt of a carbinol. Therefore, alternative routes using, among others, copper, cadmium, and zinc reagents have been employed for the syntheses of ketones from acid chlorides. In one patent, the use of $Mg(C_6F_5)X$ as an arylating reagent is claimed [7]. However, for introducing a pentafluorophenyl group, most frequently CuC_6F_5 compounds have been used [8–11]. In one paper, it has been pointed out that extensive heating (165 °C for 18 h) is necessary to convert PhCOCl into $PhCOC_6F_5$ [11]. In the presence of strong nitrogen bases, perfluoroorganophenones were prepared from 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 [12], C_6F_5 [13]) and the corresponding acid chlorides together with a number of by-products. Also the reactions of $Me_3SnC_6F_5$ transform acid chlorides into pentafluorophenones in the presence of a palladium catalyst [14]. Nickel-mediated reactions of pentafluoroiodobenzene and acid chlorides offer an alternative route to the corresponding ketones in moderate yields [15]. Reactions using $Me_3SiC_6F_5$ as an arylating reagent work in principle with these systems; unfortunately high amounts of an anhydrous fluoride source (e.g. CsF, $[NMe_4]F$) must be used to transfer the acid chloride into the corresponding fluoride prior to use [16]. In the case of reactions with Me_3SiCF_3, acid chlorides, anhydrides or activated esters can be used to prepare the corresponding bis(trifluoromethyl)carbinols via the intermediately formed trifluoromethyl ketone [17]. Reactions of LiC_6F_5 [18], $Mg(C_6F_5)X$ [19] or $Me_3SiC_6F_5$ (in the presence of fluoride) [20] with themselves give product mixtures of 1H-perfluorinated polyphenyls, $C_6F_5(C_6F_4)_nH$.

☆ For Part 1 see Ref. [2].

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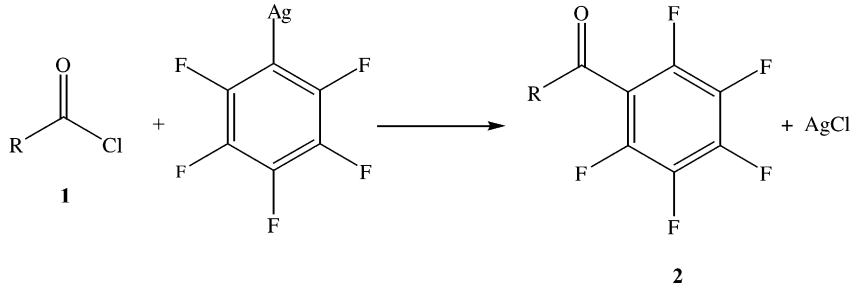


R = 4-NO_2 (a), 2-F (b), 4-F (c), 4-Cl (d), 4-Br (e), 2,3,4,5,6-F_5 (f), 2-C_4H_3O (g), 2-C_4H_3S (h), trans-PhCH=CH (j)

Scheme 1.

In this paper, we present a convenient and absolutely selective method for the preparation of 2,3,4,5,6-pentafluorophenones starting from carboxylic acid chlorides and pentafluorophenylsilver under moderate conditions.

2. Results and discussion

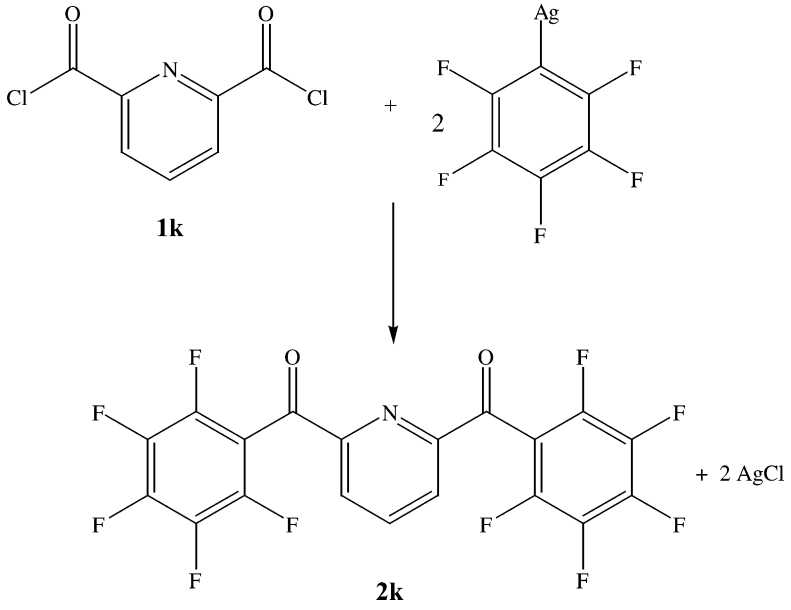
The reactions of the aroyl chlorides **1** and AgC_6F_5 proceed selectively giving the corresponding 2,3,4,5,6-pentafluorophenones **2** in good yields (Scheme 1).

In no case is there evidence for the formation of carbinols of the corresponding silver salts nor for substitution of *para*-fluorine atoms of the pentafluorophenyl group. Even in reactions with 4-ClC_6H_4COCl **1d** and 4-BrC_6H_4COCl **1e**, no NMR spectroscopic evidence for halide substitution in the 4-position has been found, although reactions were performed at elevated temperature (75–80 °C).

In a similar manner acid chlorides of furan-2-carboxylic acid **1g**, thiophene-2-carboxylic acid **1h** and *trans*-cinnamic acid **1j** were converted into the corresponding pentafluorophenones **2g–2j**.

In no case was there any evidence for by-products formation beside small amounts of C_6F_5H or acid fluoride, RCOF. The reaction principle has been extended to the acid chloride of pyridine 2,6-dicarboxylic acid **1k** (Scheme 2). The sequential C_6F_5 group transfer was monitored by ^{19}F NMR spectroscopy which showed only one characteristic set of multiplets after all AgC_6F_5 had reacted to yield 2,6-bis(2′,3′,4′,5′,6′-pentafluorobenzoyl)pyridine **2k**.

There was no NMR spectroscopic evidence for the intermediate formation of salt like species such as [RCO–N≡CEt][$Ag(C_6F_5)_2$] although perfluoroorgano silver derivatives exhibit a great tendency to form argentates [3a,b,e,21]. The probability of the formation of carbinols such as $RC(C_6F_5)_2OM$ (M = H, metal) seems to be completely suppressed due to the character of silver ions



Scheme 2.

forming complexes preferred with ''soft'' donor molecules in the sense of the well-established HSAB concept.

The reactions of carboxylic acid chlorides and pentafluorophenyl silver offer, due to the convenient accessibility of AgC_6F_5 [2], an efficient route for the selective synthesis of a series of pentafluorophenones demonstrated for ten selected examples. Further investigations with aliphatic and perfluoroaliphatic carboxylic acid chlorides and pentafluorophenyl silver as well as trifluoromethyl silver are in progress.

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 AC 200 (^{1}H, 200.1 MHz; ^{19}F, 188.3 MHz; ^{13}C, 50.3 MHz) spectrometer in $CDCl_3$ solutions. External standards were used in all cases (^{1}H, ^{13}C: Me_4Si; ^{19}F: CCl_3F). Due to the complex nature of $(AB)_2C$ spin systems in the ^{19}F NMR spectra, multiplicities are denoted as m for F-2,6 and F-3,5, respectively, tt for F-4 (pseudo-first order) [22]. Signals of the pentafluorophenyl groups occur in the $^{13}C\{^1H\}$ NMR spectra as doublets of multiplets (C-2′,6′, C-3′,5′, C-4′) with absolute values of $^1J_{F,C}$ couplings of 255 ± 5 Hz and are overlapping in most cases. As a consequence, multiplicity is given as dm without mentioning any further coupling. Shifts for C-2′,6′ and C-3′,5′ may be interchangeable. The C-1′ signal exhibits a broadened triplet with a $^2J_{F,C}$ coupling of approximately 19 ± 2 Hz; multiplicity is given as t. Assignment of all resonance for 2-fluorophenyl(2′,3′,4′,5′,6′-pentafluorophenyl)ketone was made on the assumption that similar shifts and couplings are found as determined for N-(2-fluorophenyl)-*N*′,*N*′-diethylthio urea [23]. 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. EI mass spectra were run on a Finnigan MAT 95 spectrometer (20 eV). Intensities are referenced to the most intense peak of a group. Visible melting points were determined using HWS Mainz 2000 or Stuart melting point apparatus SMP10. C, H and N analyses were carried out with HEKAtech Euro EA 3000 apparatus. Acid chlorides were prepared according to standard methods from the corresponding acids, either by treatment with thionyl chloride or oxalyl chloride. All compounds are characterized by melting points, elemental analyses, NMR spectroscopic and mass spectrometric data. For compounds described earlier values of elemental analyses are omitted.

3.1. General procedure

To a well stirred mixture of AgF (0.26 g, 2.05 mmol) in 5 ml of propionitrile $Me_3SiC_6F_5$ (0.57 g, 2.09 mmol) was added at room temperature [2]. After stirring for 1 h, the formation of AgC_6F_5 was complete. The corresponding acid chloride (2.0 mmol) was added and the reaction mixture was stirred for up to 4 h at ambient temperature and for additional 12 h at 75–80 °C. The reaction was terminated after signals of AgC_6F_5 were no longer detected in the ^{19}F NMR spectra. The complete mixture was cooled to 0 °C and poured into a 2% aqueous ammonium chloride solution with crushed ice. The organic compounds were extracted with diethyl ether. The extract was washed with water and a 5% aqueous Na_2CO_3 solution. Finally, the organic layer was dried over $MgSO_4$. Ether was evaporated and the products were crystallised from the solvents or solvent mixtures mentioned below.

3.1.1. 4-Nitrophenyl(2′,3′,4′,5′,6′-pentafluorophenyl)ketone (**2a**)

Yellow crystals. Yield: 0.60 g (95%); m.p. 123–124 °C (benzene), lit. m.p. 122–123 °C [14]. ^{19}F NMR, δ (ppm): −139.4 (m, 2F, F-2′,6′), −148.3 (tt, 1F, F-4′), −159.2 (m, 2F, F-3′,5′); ^{1}H NMR, δ (ppm): 8.01 (''d'', 2H, H-3,5, $^2J_{HH} \approx 8$ Hz); 8.35 (''d'', 2H, H-2,6, $^2J_{HH} \approx 8$ Hz). $^{13}C\{^1H\}$ NMR, δ (ppm): 183.9 (s, C=O), 140.2 (s, C-1), 130.6 (s, C-2,6), 124.2 (s, C-3,5), 151.2 (s, C-4); 112.7 (t, C-1′), 137.7 (dm, C-2′,6′), 144.1 (dm, C-3′,5′), 143.2 (dm, C-4′). MS: *m*/*z* (rel. int.): 317 $[M]^+$ (100), 195 $[C_6F_5CO]^+$ (21), 150 $[NO_2C_6H_4CO]^+$ (94).

3.1.2. 2-Fluorophenyl(2′,3′,4′,5′,6′-pentafluorophenyl)ketone (**2b**)

Pale yellow crystals. Yield 0.71 g (82%), m.p. 31–33 °C (*n*-hexane). ^{19}F NMR, δ (ppm): −112.5 (m, 1F, 2-F), −142.0 (m, 2F, F-2′,6′), −150.3 (tt, 1F, F-4′), −160.8 (m, 2F, F-3′,5′); ^{1}H NMR, δ (ppm): 7.91 (m, 1H); 7.62 (m, 1H), 7.29 (m, 1H); 7.14 (m, 1H,); $^{13}C\{^1H\}$ NMR, δ (ppm): 181.5 (C=O) 136.5 (d, $^2J_{F,C} \approx 9$ Hz,C-1), 162.0 (d, $^1J_{F,C} = 258$ Hz, C-2), 116.8 (d, $^2J_{F,C} = 22$ Hz, C-3), 125.1 (d, $^3J_{F,C} \approx 10$ Hz, C-4), 124.9 (d, $^4J_{F,C} \approx 3.5$ Hz Hz, C-5), 131.0 (s, C-6); 115.8 (t, C-1′), 137.5 (dm, C-2′,6′), 144.1 (dm, C-3′,5′), 142.6 (dm, C-4′). MS: *m*/*z* (rel. int.): 290 $[M]^+$ (100), 195 $[C_6F_5CO]^+$ (14), 123 $[FC_6H_4CO]^+$ (82). Anal. Calcd. for $C_{13}H_4F_6O$: C, 53.8; H, 1.4. Found: C, 54.2; H, 1.9.

3.1.3. 4-Fluorophenyl(2′,3′,4′,5′,6′-pentafluorophenyl)ketone (**2c**)

Colourless crystals. Yield 0.67 g (77%), m.p. 58–60 °C (*n*-hexane). ^{19}F NMR, δ (ppm): −101.5 (m, 1F, 4-F), −140.2 (m, 2F, F-2′,6′), −150.4 (tt, 1F, F-4′), −159.9 (m, 2F, F-3′,5′); ^{1}H NMR, δ (ppm): 8.72 (m, 2H, H-2,6), 7.20 (m, 2H, H-3,5); ^{13}C NMR, δ (ppm): 183.6 (s, C=O), 124.9 (d, $^4J_{F,C} \approx 2$ Hz, C-1), 132.5 (d, $^3J_{F,C} \approx 10$ Hz, C-2,6), 116.4 (d, $^2J_{F,C} \approx 22$ Hz, C-3,5), 166.4 (d, $^1J_{F,C} = 259$ Hz, C-4); 113.6 (t, C-1′), 137.7 (dm, C-2′,6′), 143.7 (dm, C-3′,5′), 142.6 (dm, C-4′). MS: *m*/*z* (rel. int.): 290 $[M]^+$ (100), 195 $[C_6F_5CO]^+$ (10), 123 $[FC_6H_4CO]^+$ (80). Anal. Calcd. for $C_{13}H_4F_6O$: C, 53.8; H, 1.4. Found: C, 54.0; H, 1.8.

*3.1.4. 4-Chlorophenyl(2′,3′,4′,5′,6′-pentafluorophenyl)ketone (**2d**)*

Colourless crystals. Yield 0.77 g (84%), m.p. 62–64 °C (*n*-hexane), lit. m.p. 63–63.5 °C [15]. ^{19}F NMR, δ (ppm): −140.0 (m, 2F, F-2′,6′), −150.0 (tt, 1F, F-4′), −159.8 (m, 2F, F-3′,5′), ^{1}H NMR, δ (ppm): 7.78 (d, $^{3}J_{H,H}$ = 8.8 Hz, 2H, H-2,6), 7.49 (d, $^{3}J_{H,H}$ = 8.8 Hz, 2H, H-3,5); ^{13}C {^{1}H} NMR, δ (ppm): 184.0 (s, C=O), 141.7 (s, C-1), 130.9 (s, C-2,6), 129.4 (s, C-3,5), 134.7 (s, C-4); 113.4 (t, C-1′), 137.6 (dm, C-2′,6′), 143.8 (dm, C-3′,5′), 142.6 (dm, C-4′). MS: *m*/*z* (rel. int.): 306 $[M]^+$ (100), 195 $[C_6F_5CO]^+$ (12), 123 $[FC_6H_4CO]^+$ (10). Anal. Calcd. for $C_{13}H_4ClF_5O$: C, 50.9; H, 1.3. Found: C, 50.8; H, 1.2.

*3.1.5. 4-Bromophenyl(2′,3′,4′,5′,6′-pentafluorophenyl)ketone (**2e**)*

Pale yellow solid. Yield 1.00 g (71%), m.p. 87–88 °C (*n*-hexane). ^{19}F NMR, δ (ppm): −139.9 (m, 2F, F-2′,6′), −149.9 (tt, 1F, F-4′), −159.7 (m., 2F, F-3′,5′); ^{1}H NMR, δ (ppm): 7.7 (“s”, 2H, H-2,6), 7.6 (“s”, 2H, H-3,5); ^{13}C{^{1}H} NMR, δ (ppm): 184.2 (s, C=O), 134.7 (s, C-1), 132.4 (s, C-2,6), 130.9 (s, C-3,5), 130.6 (s, C-4); 113.4 (t, C-1′), 137.7 (dm, C-2′,6′), 143.8 (dm, C-3′,5′), 142.6 (dm, C-4′). MS: *m*/*z* (rel. int.): 350 $[M]^+$ (100), 195 $[C_6F_5CO]^+$ (10), 183 $[C_6F_5O]^+$ (80), 155 $[C_6H_4Br]^+$ (10). Anal. Calcd. for $C_{13}H_4BrF_5O$: C, 44.4; H, 1.1. Found: C, 44.7; H, 1.2.

*3.1.6. Decafluorobenzophenone (**2f**)*

Colourless solid. Yield 0.49 g (68%), m.p. 91–92 °C (*n*-hexane), lit. m.p. 94 °C [24]. ^{19}F NMR, δ (ppm): −141.3 (m, 4F, F-2,6); −145.7 (tt, 2F, F-4); −160.1 (m, 4F, F-3,5); ^{13}C NMR, δ (ppm): 175.8 (s, C=O), 114.1 (t, C-1′), 145.1 (dm, C-2′,6′), 137.7 (dm, C-3′,5′), 144.1 (dm, C-4′).

*3.1.7. 2-Furyl(2′,3′,4′,5′,6′-pentafluorophenyl)ketone (**2g**)*

Orange oil. ^{19}F NMR, δ (ppm): −140.6 (m, 2F, F-2′,6′), −150.5 (tt, 1F, F-4′), −160.6 (m, 2F, F-3′,5′), ^{13}C {^{1}H} NMR, δ (ppm): 171.6 (s, C=O), 151.9 (s, C-2), 121.7 (s, C-3), 113.3 (s, C-4), 148.9 (s, C-5); 112.7 (broad, C-1′), 137.5 (dm, C-2′,6′), 143.9 (dm, C-3′,5′), 142.6 (dm, C-4′). The sample was contaminated by up to 10% of furan-2carboxylic acid.

*3.1.8. 2-Thienyl(2′,3′,4′,5′,6′-pentafluorophenyl)ketone (**2h**)*

Yellow waxy solid. Yield 0.47 g (84.5%), m.p. 20–22 °C (*n*-hexane). ^{19}F NMR, δ (ppm): −140.3 (m, 2F, F-2′,6′), −150.6 (tt, 1F, F-4′), −160.1 (m, 2F, F-3′,5′), ^{1}H NMR, δ (ppm): 7.84 (m, 1H), 7.51 (m, 1H), 7.17 (m, 1H); ^{13}C{^{1}H} NMR, δ (ppm): 176.5 (s, C=O), 142.9 (s, C-2), 136.1 (s, C-3), 137.3 (s, C-4) 128.7 (s, C-5); 113.9 (t, C-1′), 137.5 (dm, C-2′,6′), 143.6 (dm, C-3′,5′), 142.4 (dm, C-4′). MS: *m*/*z* (rel. int.): 278 $[M]^+$ (100), 195 $[C_6F_5CO]^+$ (22), 111 $[C_4H_3SCO]^+$ (40), 83 $[C_4H_3S]^+$ (16). Anal. Calcd. for $C_{11}H_3F_5OS$: C, 47.5; H, 1.1; S, 11.5. Found: C, 47.1; H, 0.9; S, 11.7.

*3.1.9. trans-Styryl(2′,3′,4′,5′,6′-pentafluorophenyl)ketone (**2j**)*

Colourless to pale yellow crystals. Yield 0.51 g (85%), m.p. 102–103 °C (benzene:hexane = 1:1), lit. m.p. 102–103 °C [8]. ^{19}F NMR, δ (ppm): −140.9 (m, 2F, F-2′,6′), −150.6 (tt, 1F, F-4′), −160.3 (m, 2F, F-3′,5′); ^{1}H NMR, δ (ppm): 7.56 (m, 3H, phenyl), 7.52 (d, $^{3}J_{H,H}$ = 16.6 Hz, 1H, =CH); 7.44 (m, 2H, phenyl), 7.02 (d, $^{3}J_{H,H}$ = 16.6 Hz, 1H, =CH); ^{13}C{^{1}H} NMR, δ (ppm): 183.8 (s, C=O), 148.1 (s, PhC=), 126.0 (s, =CCO); 133.5 (s, C-1), 129.1 (s, C-2,6), 128.9 (s, C-3,5), 131.7 (s, C-4); 114.5 (t, C-1′), 137.6 (dm, C-2′,6′), 144.1 (dm, C-3′,5′), 142.6 (dm, C-4′). MS: *m*/*z* (rel. int.): 298 $[M]^+$ (78), 297 $[M–H]^+$ (100), 195 $[C_6F_5CO]^+$ (4), 131 $[C_9H_7O]^+$ (15), 103 $[C_8H_7]^+$ (12).

*3.1.10. 2,6-Bis(2′,3′,4′,5′,6′-pentafluorobenzoyl)pyridine (**2k**)*

Pale yellow crystals. Yield 0.51 g (54%), m.p. 164–166 °C (*n*-hexane:benzene = 1:1). ^{19}F NMR, δ (ppm): −139.1 (m, 2F, F-2′,6′), −149.3 (tt, 1F, F-4′), −160.5 (m, 2F, F-3′,5′); ^{1}H NMR, δ (ppm): 8.41 (“m”, 2H, H-3,5), 8.14 (“t”, 1H, H-4); ^{13}C{^{1}H} NMR, δ (ppm): 185.0 (s, C=O), 151.4 (s, C-2,6), 127.3 (s, C-3,5), 139.3 (s, C-4); 113.0 (t, C-1′), 137.1 (dm, C-2′,6′), 144.1 (dm, C-3′,5′), 142.6 (dm, C-4′). MS: *m*/*z* (rel. int.): 467 $[M]^+$ (28), 439 $[M–CO]^+$ (86), 411 $[M–2CO]^+$ (100), 195 $[C_6F_5CO]^+$ (36). Anal. Calcd. for $C_{19}H_3F_{10}NO_2$: C, 48.8; H, 0.6; N 3.0. Found: C, 49.4; H, 0.7; N, 3.7. The analytical data and low intensity signals in the NMR spectra indicated the monosubstituted product 2-(C_6F_5CO)-6-COOH-C_5H_3N as an impurity.

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