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Reaction of 2,3,4,5,6-Pentafluorobenzamide with Potassium Hydride: Unexpected Activation of the C–F Bond and Dimerization of Organofluorine Ligand

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Abstract—The reaction of potassium hydride with 2,3,4,5,6-pentafluorobenzamide (FBAm) in dimethoxyethane results in activation of the C–F bond in the *para*-position to the $C(O)NH_2$ group followed by dimerization of FBAm to form a potassium salt with one free amide group. The structure of the binuclear reaction product $\{(DME)_2K^+[C_6F_5–C(O)N–C_6F_4–C(O)NH_2]^-\}_2$ (**I**) was determined by X-ray diffraction (CCDC 2311402), the purity of the product was confirmed by NMR spectroscopy.

Keywords: carboxylic acid amides, pentafluorobenzamide, potassium hydride, C–F bond activation, X-ray diffraction

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INTRODUCTION

Fluorinated organic compounds are of considerable interest for materials chemistry, pharmaceutical chemistry, agrochemistry, organic chemistry, and polymer chemistry [1–6]. Representatives of this class of compounds are successfully used as anticancer, antioxidant, fungicidal, and other agents possessing a wide range of biological activities [7–10]. On the other hand, compounds with fluorine-containing groups are promising reagents for the preparation of diverse classes of organic products [11]. Therefore, the development of efficient methods for the synthesis of organofluorine compounds and methods for C–F bond activation are the important tasks of the modern chemistry and materials science [12–18].

Currently, numerous catalytic and non-catalytic reactions involving compounds of transition metals, particularly platinum group metals, for C–F bond activation are known. This can be exemplified by cross-coupling reactions of fluorinated aromatic derivatives catalyzed by compounds of nickel [19–21], palladium [22–24], ruthenium [25], and other metals; C–F bond cleavage with insertion of a transition metal derivative [26–29]; catalytic hydrodefluorination [30–33], and many other reactions [5]. Meanwhile, quite a few papers are devoted to C–F bond activation by nonmetal and main group metal compounds without participation of transition metals (a large array of studies is addressed in detail in a review [17]). The insertion of main group metal compounds into the C–F bond and substitution of fluorine in aromatic fluorides either in the presence of rhodium or nickel catalysts or without an additional catalyst were considered in [18]. A number of publications describe the reductive catalytic hydrodefluorination of fluorobenzenes with metal hydrides (NaH, $LiAlH_4$) in the presence of minor amounts of transition metal salts [34–36] and S_NAr aromatic substitution reactions with replacement of fluorine by oxygen-, sulfur-, and carbon-containing nucleophilic groups on treatment with sodium hydride to give various functional (including heterocyclic) derivatives [37–42].

In this study, we demonstrated that the reaction of pentafluorobenzamide with potassium hydride in dimethoxyethane is also accompanied by substitution of the fluorine atom in the *para*-position of the benzene ring of one pentafluorobenzamide molecule by the pentafluorobenzamide group of the other molecule to give the potassium salt $\{(DME)_2K^+[C_6F_5–C(O)N–C_6F_4–C(O)NH_2]^-\}_2$ (**I**).

EXPERIMENTAL

All operations on the synthesis of compound **I** were carried out under inert atmosphere using the standard Schlenk technique. A commercial suspension of potassium hydride in mineral oil (Aldrich) was pretreated with dehydrated hexane in order to isolate KH powder, which was stored and weighed in a glovebox. Dimethoxyethane (DME, Acros) was dried with

sodium metal, stored over sodium benzophenone ketyl, and transferred by vacuum condensation immediately before the reaction; 2,3,4,5,6-pentafluorobenzamide (98%, P&M Invest) was used as received. The IR spectrum of compound **I** was recorded in the 400–4000 cm^{-1} range by the attenuated total reflectance (ATR) method on a Perkin Elmer Spectrum 65 spectrophotometer equipped with a Quest ATR Accessory attachment (Specac). ^1H and ^{19}F NMR spectra were measured on a Bruker AVANCE-300 spectrometer operating at 300 MHz with tetramethylsilane as an internal standard and DMSO-d_6 as a solvent.

Synthesis of $\{(DME)_2K^+[C_6F_5–C(O)N–C_6F_4–C(O)NH_2]^-\}_2$ (I). 2,3,4,5,6-Pentafluorobenzamide (0.211 g, 1 mmol) was placed into a glass tube and evacuated for 20 min under dynamic vacuum. Then DME (15 mL) was condensed into the tube, and the solution was cooled by liquid nitrogen, while keeping the reaction mixture from freezing. Potassium hydride (0.040 g, 1 mmol) was added in portions to control the temperature of the reaction mixture and the flow of hydrogen. After addition of the whole amount of potassium hydride and completion of gas release (2 h), the reaction mixture was a slightly yellowish transparent solution. The subsequent concentration (to 5 mL) and keeping of the solution at 6°C (24 h) gave crystals suitable for X-ray diffraction. The mother liquor was decanted, and the product was washed with cold DME. The yield of the crystalline product **I** was 0.113 g (34%).

For $C_{96}H_{104}N_8O_{28}F_{36}K_4$		
Anal. calcd., %	C, 43.38	H, 3.94
Found, %	C, 43.27	H, 3.80

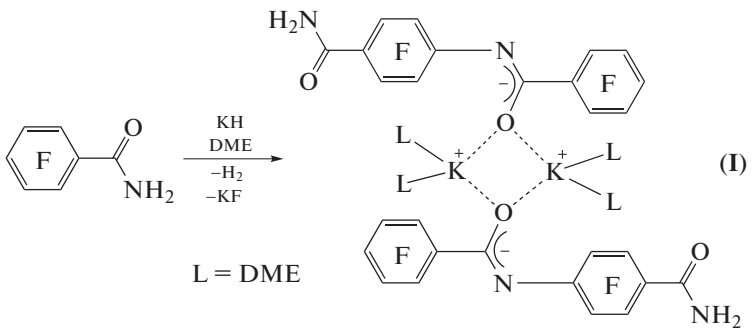
IR (ν, cm^{-1}): 3247 w, 3002 w, 2937 w, 2899 w, 2836 w, 2727 w, 1684 m, 1650 m, 1566 s, 1517 m, 1478 s, 1407 s, 1363 s, 1289 w, 1256 m, 1196 m, 1124 m, 1090 s, 1062 s, 1033 m, 988 vs, 941 m, 904 m, 853 m, 795 m, 742 m, 652 m, 570 w, 486 m, 438 w, 409 w. ^1H NMR (300 MHz; DMSO-d_6; δ, ppm): 3.22 (s, 12H, CH_3), 3.42 (s, 8H, CH_2), 7.81 (s, 1H, NH_2), 8.11 (s, 1H, NH_2). ^{19}F NMR (282 MHz; DMSO-d_6; δ, ppm): –165.03 (m, 2F), –161.55 (m, 1F), –151.42 (m, 2F), –148.87 (m, 2F), 145.19 (m, 2F).

The single-crystal X-ray diffraction study of compound **I** was performed on a Bruker APEX II diffractometer (CCD detector, MoK_α radiation, λ = 0.71073 Å, graphite monochromator) [43]. A semiempirical absorption correction was applied [44]. Using the Olex2 software [45], the structure was solved on the basis of unique reflections of a domain of **I** with ShelXT [46] and refined with hkl5 program with the olex2.refin refinement package [45] using least-squares minimization on F^2 in the anisotropic approximation for non-hydrogen atoms. The hydrogen atoms at the carbon atoms of organic ligands were generated geometrically and refined in the riding model. The calculations were carried out by means of the OLEX2 program package [45]. The structure was refined with allowance for the disorder of one coordinated and solvation dimethoxyethane molecule using standard ISOR, DFIX, and EADP constraints. Crystallographic data and structure refinement details for **I**·2DME are as follows: $C_{96}H_{104}F_{36}K_4N_8O_{28}$, M = 2658.27; 0.15 × 0.12 × 0.09 mm crystal; T = 120(2) K, triclinic system, space group $P\bar{1}$, a = 11.745(8), b = 12.516(8), c = 22.832(14) Å, α = 97.731(13)°, β = 90.291(14)°, γ = 116.601(14)°, V = 2966(3) $Å^3$, Z = 1, ρ = 1.488 g/cm^3, μ = 0.280 mm^{-1}, θ = 1.81°–25.00°, $-13 \le h \le 13$, $-14 \le k \le 14$, $0 \le l \le 27$; 19 101 reflections were measured, 10 167 reflections were unique, 7394 reflections had $I \ge 2\sigma(I)$, R_{int} = 0.0460, T_{min}/T_{max} = 0.2752/0.3813, GOOF = 1.048, R_1 = 0.1002, wR_2 = 0.2597 (for $I \ge 2\sigma(I)$), R_1 = 0.1281, wR_2 = 0.2812 (for all data), $\Delta\rho_{min}/\Delta\rho_{max}$ = –0.904/0.962 e $Å^{-3}$.

The crystallographic parameters for the structure of **I** were deposited with the Cambridge Crystallographic Data Centre (CCDC no. 2311402; deposit@ccdc.cam.ac.uk; http://www.ccdc.cam.ac.uk).

RESULTS AND DISCUSSION

The reaction of potassium hydride with perfluorobenzamide in dimethoxyethane not only gives the perfluorobenzamide potassium salt, but also activates the fluorine–carbon bond in the *para*-position of the aryl group, resulting in the formation of dimeric product **I** with one free amide group (Scheme 1).



Scheme 1.

Table 1. Selected bond lengths (Å) and bond angles (deg) in the structure of **I**

Bond	Molecule A	Molecule B	Angle	Molecule A	Molecule B
	d, Å			ω, deg	
K–O(L)	2.722(4), 2.739(4)	2.696(5), 2.756(5)	K(1)O(5)K(1)	96.79(13)	94.27(13)
K–O(DME)	2.720(4)–2.868(5)	2.723(4)–2.854(5)	O(5)K(1)O(5)	83.21(13)	85.73(13)
C(5)–O(5)	1.235(7)	1.255(7)	O(5)C(5)C(6)	118.1(5)	118.1(5)
C(22)–O(6)	1.229(7)	1.220(7)	O(5)C(5)N(1)	130.5(5)	130.7(5)
C(5)–N(1)	1.310(8)	1.324(8)	C(5)N(1)C(12)	117.6(5)	116.5(5)
N(1)–C(12)	1.405(7)	1.398(7)	O(6)C(22)N(2)	125.1(5)	125.5(5)
C(22)–N(2)	1.314(8)	1.310(8)	O(6)C(22)C(15)	119.2(5)	118.8(5)
C(5)–C(6)	1.524(8)	1.531(7)	N(2)C(22)C(15)	115.7(5)	115.7(5)
C(22)–C(15)	1.511(7)	1.508(8)			

Compound **I** was isolated in the crystalline state and characterized by ^{1}H and ^{19}F NMR and IR spectroscopy and X-ray diffraction analysis.

In the ^{1}H NMR spectrum of compound **I**, the $C(O)NH_2$ protons are manifested as two singlets at 7.81 and 8.11 ppm (DMSO-d_6) which are shifted upfield compared to those for the initial pentafluorobenzamide (8.16 and 8.31 ppm, [47]); the spectrum also exhibits signals for dimethoxyethane protons (singlets at 3.22 and 3.42 ppm). According to ^{19}F NMR data, compound **I** contains five non-equivalent groups of fluorine atoms with an intensity ratio of: 2 : 2 : 2 : 2 : 1, which is in line with structural data.

According to X-ray diffraction data, **I** crystallizes in the triclinic space group $P\bar{1}$ with two centrosymmetric independent $[K_2L_2(DME)_4]$ molecules (molecules A and B, Fig. 1) and two DME solvation molecules in the crystal cell. Regarding the structure, bond lengths, and bond angles (Table 1), $[K_2L_2(DME)_4]$ molecules are similar; hence the geometry can be considered for one of them. The inversion center is located between the potassium atoms (K...K, 4.083(3) and 3.996(3) Å; hereinafter, for molecules A and B, respectively), each of them coordinating two chelating DME molecules and two bridging oxygen atoms of the two C_6F_5–C(O)N– moieties of L. The K–O bond lengths with the bridging oxygen atoms vary in the range of 2.696(5)–2.756(5) Å. In ligand L, the bond lengths in the C_6F_5–C(O)N– moiety (C=O, 1.235(7) and 1.255(7) Å; C–N, 1.310(8) and 1.324(8) Å) and in the amide moiety (C=O, 1.229(7) and 1.220(7) Å; C–N, 1.314(8) and 1.310(8) Å) correspond to the known literature data. The angles between C(O)N and the C_6F_5 ring are 61.5(5)° and 66.7(5)°; the angles with the C_6F_4 ring are 72.3(4)° and 64.5(6)°; and the angles between the amide moiety and the C_6F_4 ring are 82.4(5)° and 70.0(5)°. The protons of the coordinated DME molecules in $[K_2L_2(DME)_4]$ are involved in the C–H...π, C=O...π, C–H...O, and C–H...F intramolecular contacts (Tables 2, 3).

The crystal packing of **I** is determined by a set of intermolecular non-valent contacts, including π-stacking interactions between the tetrafluoro-substituted phenyl aromatic rings (Table 4) and the C=O...π, N–H...O, and N–H...N interactions (Table 2, 3). The intermolecular interactions of the aromatic rings and the H-bonds between the C_6F_5–C(O)N– nitrogen atom and the amide group give rise to supramolecular chains (Fig. 2). The chains of molecules A are aligned along the [1 1 0] vector, and molecules B are arranged along the [0 1 0] vector or the 0*b* axis; the angle between these vectors is 55.4°. The chains form sublattices of layers parallel to the 0*ab* plane via Van der Waals interactions, including weak C–H...F contacts (H...F, 2.49 and 2.63 Å). Every two chains of neighboring sublattices are linked to each other by two H-bonds between the amide groups and by C=O...π contacts (Fig. 2). As a result, each molecule of **I** is involved in a supramolecular chain which is bound to neighboring noncollinear chains, thus forming layers connected into a three-dimensional network.

It is noteworthy that the ability of potassium salts such as C_6F_5–XK to activate C–F bonds has been known for quite a long time. An early example of this activation is probably the synthesis of poly(tetrafluorophenylene sulfide) $(C_6F_4S)_n$ by heating potassium pentafluorothiophenolate C_6F_4–SK in a sealed evacuated tube at a temperature of 240°C for three hours [48, 49]. The presumptive mechanism of fluorine atom substitution in FBAm to form the dimer is presented in Scheme 2. During the reaction, we observed the hydrogen evolution; this suggests that the first step leads to the formation of the monopotassium salt of pentafluorobenzamide which further reacts with free perfluorobenzamide to give weakly bound intermediate Int-1. The negative charge transfer in Int-1 to the perfluorinated ring of pentafluorobenzamide gives Int-2. The charge and bond redistribution in Int-2 giving off a proton and a fluoride anion in the presence

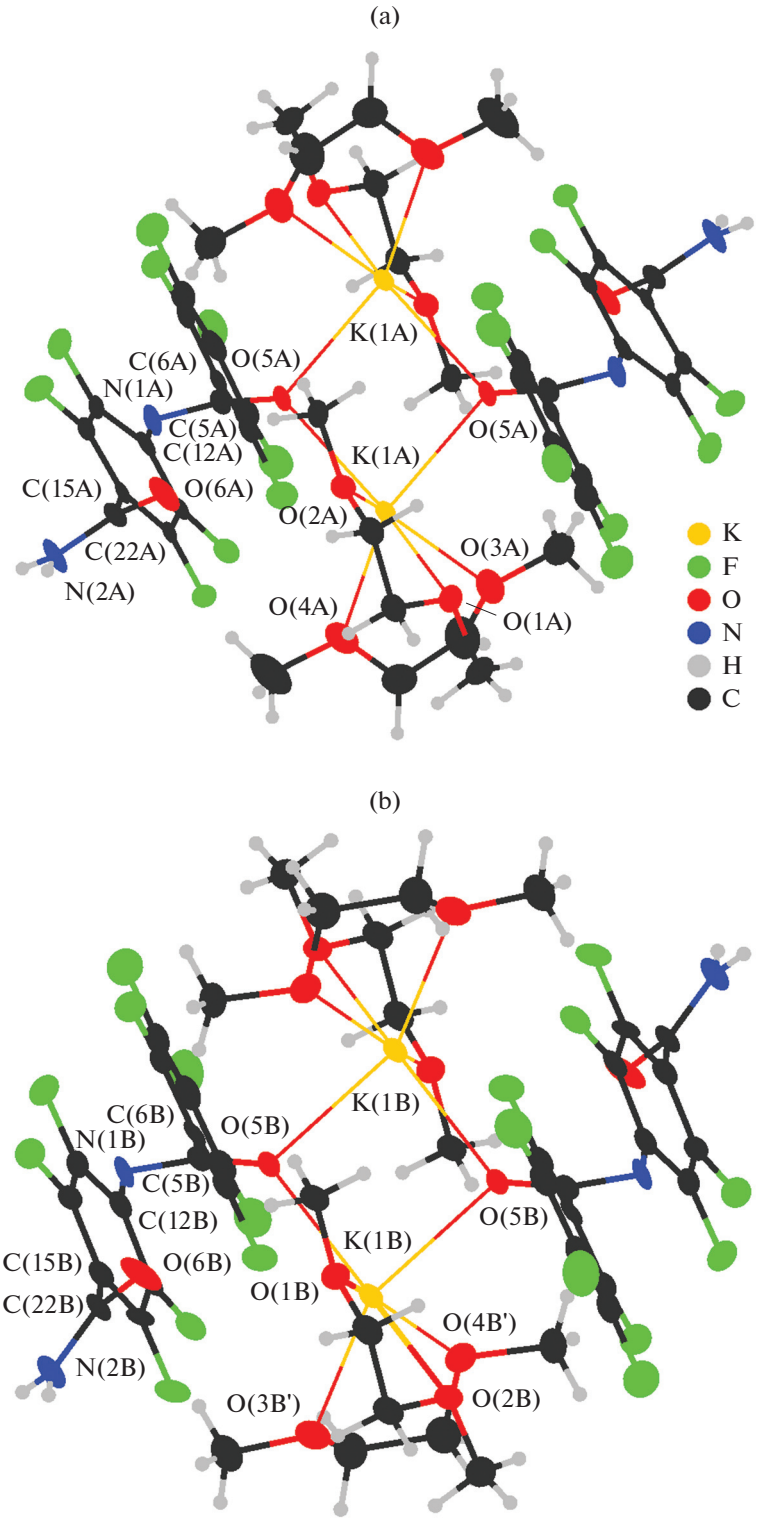


Fig. 1. Molecular structures of two independent molecules (a) A and (b) B in compound **I** (thermal ellipsoids are given at 30% probability; the solvation molecules and disordered DME molecules in molecule A are not shown).

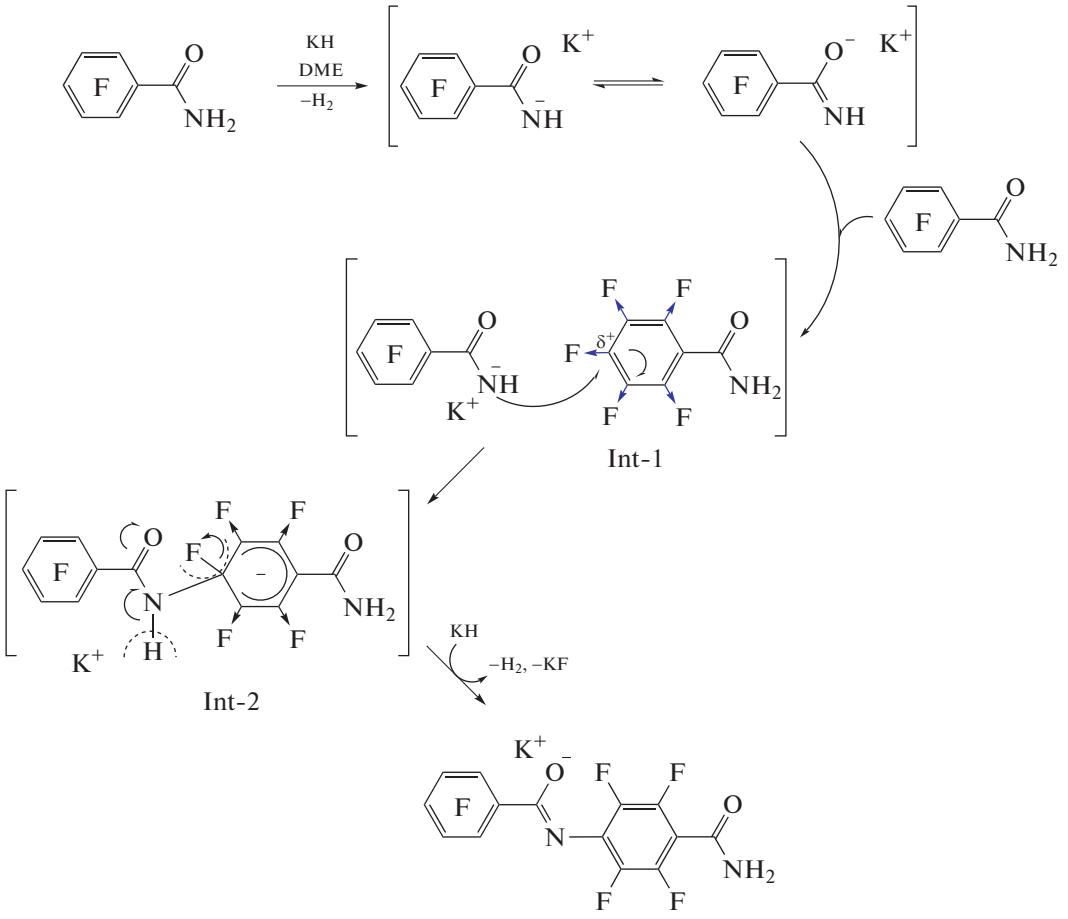
of a second KH molecule results in hydrogen evolution and formation of potassium fluoride and gives the final product **I**. This mechanism can be considered as aromatic nucleophilic substitution S_NAr of a fluorine atom in the aromatic ring which is described in a number of publications [5, 37–42].

Table 2. Parameters of the C–Y···π interactions in the crystal packing of **I** (Cg_i is the centroid of the aromatic ring; Y–Perp is the shortest distance from atom Y to the ring plane: Cg_1–C(6B) → C(11B), Cg_2–C(12B) → C(17B), Cg_3–C(6A) → C(11A), Cg_4–C(12A) → C(17A)

Contact	Y···Cg, Å	Y–Perp, Å	γ, deg	C–Y···Cg, deg	C···Cg, Å
C(1B)–H(1BA)...Cg_1 (–*x*, 2 – *y*, –*z*)	2.86	2.75	16.39	143	3.707(9)
C(2A)–H(2AD)...Cg_3 (1 – *x*, 2 – *y*, 1 – *z*)	2.81	2.69	16.82	144	3.662(8)
C(18B)–H(18C)...Cg_2	3.00	2.87	16.61	109	3.449(8)
C(19A)–H(19E)...Cg_4	2.97	2.08	19.52	113	3.477(8)
C(22A)–O(6A)...Cg_1 (*x*, *y*, 1 + *z*)	3.470(6)	3.058	28.21	145.5(5)	4.537(7)
C(22B)–O(6B)...Cg_3	3.347(6)	2.978	27.17	162.7(5)	4.527(7)

Table 3. Parameters of the D–H···A interactions in the crystal of **I**

Hydrogen bond	D–H, Å	H···A, Å	D···A, Å	D–H···A, deg
N(2A)–H(2AA)...O(6B) (–*x*, 1 – *y*, 1 – *z*)	0.88	2.03	2.909(7)	174
N(2A)–H(2AB)...N(1A) (–*x*, 1 – *y*, 1 – *z*)	0.88	2.03	2.910(7)	173
N(2B)–H(2BA)...O(6A) (–*x*, 1 – *y*, 1 – *z*)	0.88	2.06	2.932(7)	173
N(2B)–H(2BB)...N(1B) (–*x*, 1 – *y*, –*z*)	0.88	2.08	2.926(7)	161
C(1SA)–H(1SA)...F*(8B) (–*x*, 1 – *y*, –*z*)	0.98	2.48	3.306(19)	141
C(18B)–H(18B)...O(5B)	0.98	2.57	3.377(8)	139
C(21A)–H(21I)...F(13A)	0.98	2.35	3.103(15)	132



Scheme 2.

Table 4. Parameters of the π···π interactions in the crystal packing of **I***

Contact	Cg···Cg, Å	Cg···Perp, Å	α, deg	Shift, Å
Cg_2···Cg_2 (–*x*, 1 – *y*, –*z*)	3.592(4)	3.479(2)	0	0.895
Cg_4···Cg_4 (–*x*, 1 – *y*, 1–*z*)	3.640(4)	3.473(2)	0	1.093

* Cg_i is the centroid of the benzene ring; Cg–Perp is the shortest distance from Cg to the plane of the neighboring ring; α is the angle between the planes Cg, Cg_2–C(12B) → C(17B), Cg_4–C(12A) → C17A; shift is the distance between the Cg_i centroid and projection of the Cg_j centroid onto the plane of ring *i*.

Thus, the reaction of potassium hydride with perfluorobenzamide in dimethoxyethane leads to the activation of the fluorine–carbon bond in the *para*-position and gives the dimeric product $\{(DME)_2K^+[C_6F_5–C(O)N–C_6F_4–C(O)NH_2]^-\}_2$ which was studied by X-ray diffraction, and the identity of the product was confirmed by NMR spectroscopy.

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CONFLICT OF INTEREST

The authors of this work declare that they have no conflicts of interest.

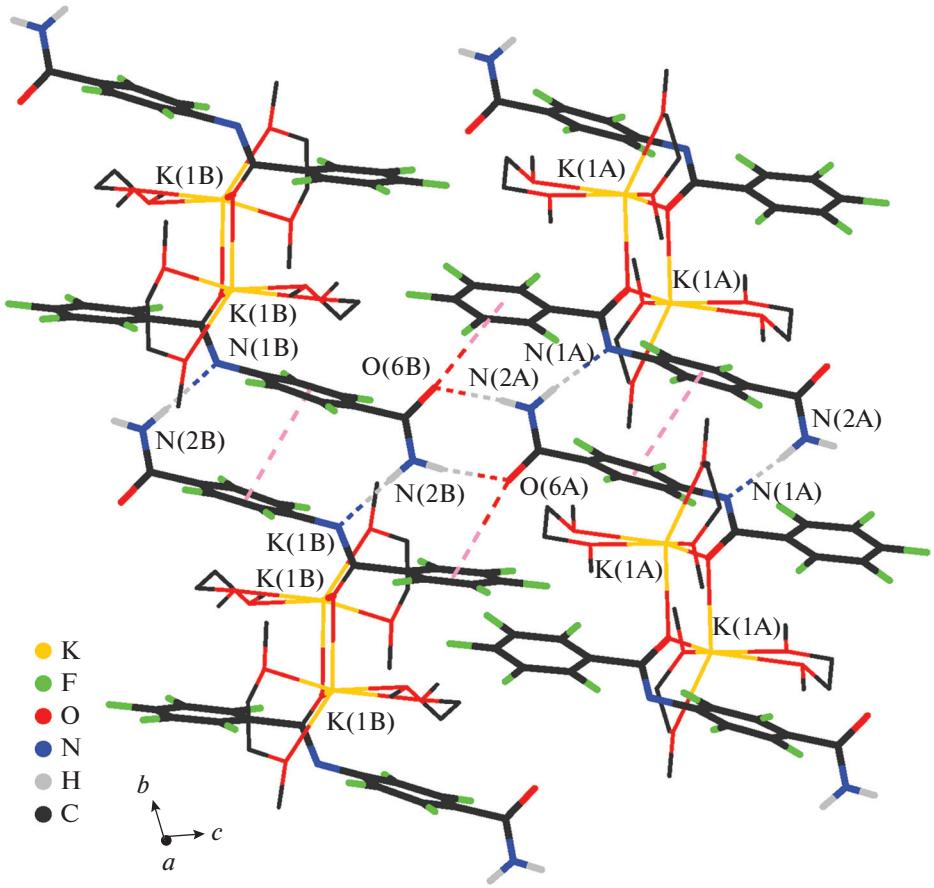

Fig. 2. Fragment of packing of **I** in the crystal (the hydrogen atoms at DME molecules and solvation molecules are not shown; H-bonds and C=O...π and π...π interactions are shown by dashed lines).

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