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Facile one-pot fluorination of polycyclic aromatic hydrocarbons (PAHs) with *N*-fluoro-2,4-dinitroimidazole; scope and limitation

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This paper is dedicated to Prof. Robert Filler

Abstract

The synthetic utility of *N*-fluoro-2,4-dinitro-imidazole 'NF-2,4-DNI', a recently introduced NF fluorinating agent, has been tested for direct one-pot fluorination of several classes of polycyclic aromatic hydrocarbons, PAHs, namely pyrene, crowded alkyl(cycloalkyl)-pyrenes; hexahydro- and tetrahydro-pyrene; benzo[a]anthracene; benzo[a]- and benzo[e]pyrene; perylene; 2,7-di-*tert*-butylphenanthrene; chrysene; 9-methylanthracene and anthracene, as well as *trans*-15,16-dimethyl-dihydropyrene; azulene; 2-acenaphthylene and azulene. Although the isolated yields are modest, the ease of handling of the reagent, simple operation (reflux in dichloroethane for 3 days) and the use of 1.1 equivalent of the reagent makes the procedure quite attractive for polynuclear aromatics, avoiding multi-step operations (NO_2–PAH → NH_2–PAH → N_2^+–PAH → F-PAH) or the use of toxic or costly reagents (CF_3OF, XeF_2, etc.); it provides direct one-pot access to a variety of F-PAHs that are not readily made using other fluorinating agents.

Keywords: One-pot fluorination; Polycyclic aromatic hydrocarbons; Fluorinating agent; Polynuclear aromatics; Pyrene

1. Introduction

From several perspectives, fluorinated polycyclic aromatic hydrocarbons, F-PAHs, continue to play an important role in elucidation of the mechanism of carcinogenesis of PAHs, for example, in narrowing down possible active sites which bind to DNA [1,2], in modulating carcinogenicity from a site 'remote' to the binding site(s) [3–6], and in conformational studies of diol-epoxides where fluorine may control bay-region diol-epoxide reactivity [7,8]. Another active area where F-PAHs have gained importance relates to the mode of action of their radical cations with model nucleophiles [9,10].

There is a continuing need to search for short synthetic routes and mild reagents to prepare F-PAHs for mechanistic studies in connection to carcinogenicity. Despite the synthetic development of numerous 'electrophilic' fluorinating agents in recent years, direct fluorine introduction into polycyclic aromatics continues to remain a challenge [9–11].

Whereas the traditional fluoro-dediazoniation strategy (decomposition of $ArN_2^+ BF_4^-$) is practical for alkylnitrobenzenes due to their ready availability, especially using HF/pyridine which gives better yields of the Baltz–Schiemann reaction [12], this approach becomes impractical for PAHs due to the need for a multi-step operation and several separations [13]. Moreover, for sterically crowded systems the diazotization step is usually very sluggish and the overall yields are at best poor. An illustrative example of the amount of work that can be involved in selective PAH fluorination, is a recent modification applied to the synthesis of 1- and 3-fluorobenzo[a]pyrene from 6-chlorobenzo[a]pyrene which involves N_2O_4 nitration, reduction ($SnCl_2 \cdot H_2O$), conversion to triazene, fluorination of triazene with HF/pyridine; HPLC separation and selective debromination [14].

Fluorination of benzenes and representative fused polycyclic aromatics with XeF_2 (and its intercalates) have been reported [15–22,42] and the topic has been reviewed [23]. Observation of addition compounds, side-chain fluorination, and formation of biphenyl and polyphenyls in these systems are indicative of an oxidative mechanism [23]. CF_3OF (a highly toxic gas) has been used for PAH fluorination in several cases leading to mixtures of monofluorination, *gem*-difluorination and oxidation products [24]; $CsSO_4F$ fluorinations of phenanthrene and pyrene are also reported [24].

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Although the 'electrophilic' NF reagents 'Selectfluor' and their derivatives are easily handled and are indeed useful for fluorination of carbanions, alkenes and some activated benzene derivatives [25,26], they offer limited utility in fluorination of polycyclic arenes [25,27]. The 1-chloromethyl-4-fluoro-1,4-diazonia-bicyclo[2.2.2]octane bis(tetrafluoroborate) 'F-TEDA-BF_4' combined with triflic acid was very recently reported to increase the scope of aromatic fluorination [28]. In our hands, all attempts to fluorinate reactive PAHs such as polyalkylpyrenes with F-TEDA-BF_4 led to intractable mixtures [29]. Fluorination of arenes with NF-sulfonimides such as *N*-fluoro-*o*-benzene-disulfonimide, NFOBS, and *N*-fluorobenzenesulfonimide, NFSi, appears to be limited to anisole; although toluene was fluorinated when a large excess (50 fold) of the NF-reagent is used [30,31]. We report here on the utility of *N*-fluoro-2,4-dinitroimidazole 'NF-2,4-DNI' in fluorination of polycyclic aromatics.

2. Results and discussion

2,4-Dinitro-imidazole synthesized by protic nitration of the commercially available 4-nitro-imidazole according to the literature [32,33], was *N*-fluorinated (5% F_2 in N_2) to give 'NF-2,4-DNI' **1**, as a white solid [34,35]. The premise was that the electron withdrawing effect of the nitro groups should weaken the NF bond, allowing transfer-fluorination to PAHs under mild conditions.

Fluorination of pyrene with **1** was carried out under various conditions in an effort to optimize the yields. The results are summarized in Table 1. Under comparable conditions, optimal results were obtained using 1,2-dichloroethane as solvent under reflux for 3 days using 1.1 mole equivalent of the reagent (Fig. 1). The yields diminished with the use of excess fluorinating agent. In these cases, the solutions were darker and less of the intact PAH could be recovered (see also later discussion).

Modeling after Olah's transfer-nitration reaction with *N*-nitro-pyrazole [36], where the use of BF_3·etherate or protic superacids enhanced transfer-nitration ability by further weakening the N–NO_2 bond (thus lowering the energy barrier), we explored both BF_3 (gas) and triflic acid in an attempt to induce transfer-fluorination at or near room temperature. In an effort to minimize complexation or protonation of the arene, **1** was pretreated with the additive and then a solution of the PAH in 1,2-dichloroethane was added. These room temperature reactions did not, however, result in any

Table 1
Fluorination of pyrene under various conditions

Solvent	Fluorination reagent (mmol)	Additive	Temp. (°C)	Yield (%)
$C_2H_4Cl_2$	0.11	none	reflux	23
$C_2H_4Cl_2$	0.15	none	reflux	20
$CHCl_3$	0.11	none	reflux	18
CH_3CN	0.11	none	reflux	14
$C_6H_5NO_2$	0.11	none	100	8
CF_3CH_2OH	0.11	none	reflux	16
$C_2H_4Cl_2$	0.11	CF_3SO_3H	reflux	0
$C_2H_4Cl_2$	0.11	BF_3	reflux	0

The reaction was carried out with 0.1 mmol of pyrene, 0.2 mmol of additive, and 20 ml of solvent for 3 days.

fluorination, suggesting that the additive complexed or protonated the more basic PAH in equilibrium hence reducing its basicity/nucleophilicity. Switching to the 'normal reflux conditions' in the presence of the additive also prevented the fluorination reaction completely.

It is pertinent to point out that the purity of **1** had little effect on the fluorination ability. Thus a raw mixture consisting of 70% **1** and 30% 2,4-dinitro-imidazole precursor exhibited nearly the same fluorination power. Formation of 2,4-dinitro-imidazole is clearly seen during slow fluorination as it slowly precipitates out of dichloroethane. For pyrene a 9:1 mixture of 1-fluoro- and 4-fluoropyrene was obtained regardless of the reaction conditions.

2.1. Survey of PAH Fluorination with 1

The yields, regioselectivities (isomer distributions) and the ^{19}F NMR data are gathered in Figs. 2–4 for a range of PAHs examined in this study. A noteworthy feature of these reactions is the formation of PAH dimers in several cases as byproducts and the diminished reactivity and eventually the lack of it, with increasing ionization potentials in the PAHs (i.e., 1-methylnaphthalene, 2-methylphenanthrene, triphenylene) [37]. These observations are more in line with a radical cation process than with electrophilic fluorination. Diminished yields, darker solutions, and reduced recovery of the intact PAH, in cases where excess fluorinating agent was used, also appear compatible with an oxidative mechanism which increases side product (tar) formation.

Benzo[a]anthracene: Fluorination occurred at the C-7/C-12 positions to give a mixture of two isomeric mono- (**5** and **6**) and the 7,12-difluorobenzo[a]anthracenes (**7**), whose rel-

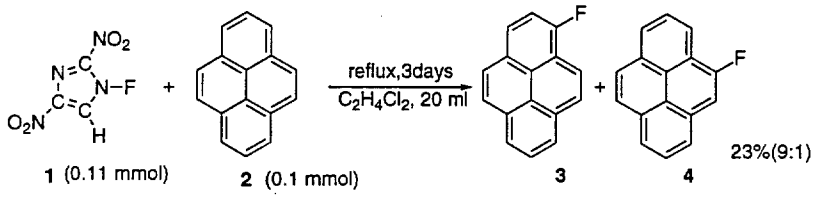

Fig. 1. Fluorination of pyrene with the NF compound **1** under optimized reaction conditions.

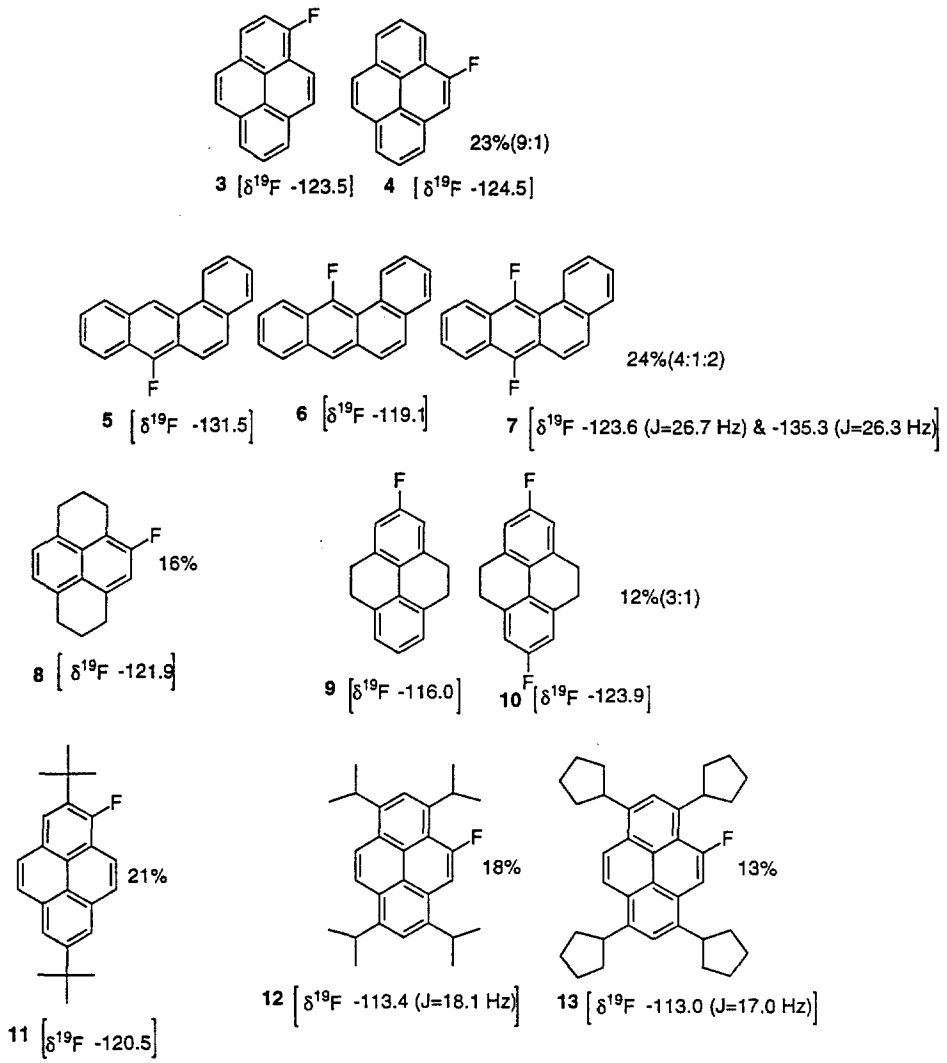

Fig. 2. Fluorination of pyrene, benz[a]anthracene, tetrahydro- and hexahydropyrene, 2,7-di-*tert*-butylpyrene, 1,3,6,8-tetraisopropyl- and 1,3,6,8-tetracyclopentylpyrene; the yields, relative isomer distributions, and the ^{19}F NMR data are shown.

ative assignments were made based on the ^{19}F NMR data in comparison with the literature [17,38,39]. Presence of a 5-bond F/F coupling in the ^{19}F NMR spectrum of **7** is noteworthy and has been mentioned before [38]. Fluorination of benzo[a]anthracene with $C_{19}XeF_6$ was reported to give only **5** [17], whereas its anodic fluorination [38] (capture of fluoride ion by the radical cation) produced **5**, **6**, and **7**. The regioselectivities (and product ratios) for fluorination with **1** closely resemble the anodic fluorination data.

1,2,3,6,7,8-Hexahydro- and 4,5,9,10-Tetrahydropyrene: Hexahydropyrene gave the monofluoro derivative **8** [19], whereas tetrahydropyrene produced a 3:1 mixture of the mono- and difluorinated derivatives (**9** [19,40] and **10**) which could not be separated; compound **10** had a shorter GC retention time as compared to **9** (Fig. 2).

2,7-di-*tert*-Butylpyrene: Fluorination of 2,7-di-*tert*-butylpyrene gave the 1-fluoro derivative **11** selectively in 21% yield (Fig. 2).

1,3,6,8-tetraisopropyl- and 1,3,6,8-tetracyclopentylpyrene: All previous attempts to synthesize the fluoro compounds of these crowded all α-substituted pyrenes had failed [29]. Utilizing reagent **1**, the desired monofluoro derivatives **12** (m/z 388) and **13** (m/z 492) were obtained in reasonable yields (Fig. 2). In the case of tetra-isopropylpyrene, apart from **12**, a fluoro-de-isopropylation product ($\delta^{19}F$ −124.3; m/z 346) as well as traces of difluoro-tri-isopropyl- (m/z 364) and difluoro-tetraisopropylpyrene (m/z 406) were identified. In the case of **13**, a trace of fluoro-dealkylation product was present (m/z 424). Vicinal H–F couplings in compounds **12** and **13** are in the 17–18 Hz range.

2,4,6,8,10-Pentaisopropylpyrene (Fig. 3): A mixture of two isomeric monofluoro compounds (**14**, **15**; m/z 430) were isolated in 5:1 ratio in addition to several side products, which based on GC-MS analysis were found to be the monofluoro-tetraisopropyl- (m/z 388), difluoro-penta-isopropyl- (m/z 448), and a trace of monofluoro-tri-isopropylpyrene (m/z 346).

Benzo[a]pyrene: Following fluorination, the products were separated by preparative HPLC. Apart from the desired 6-fluoro compound **16** [22,23], a PAH dimer was formed as

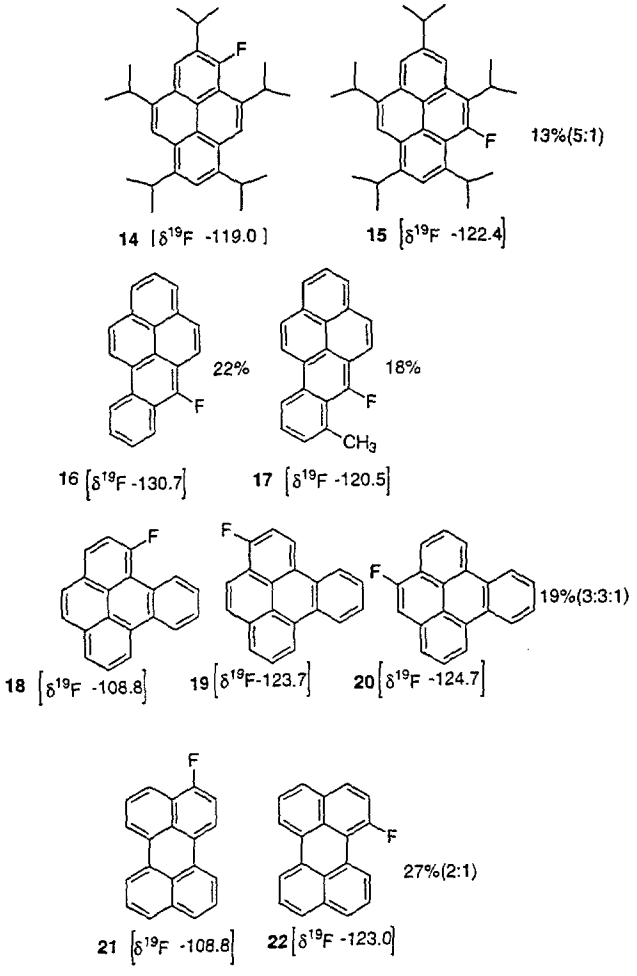

Fig. 3. Fluorination of 2,4,6,8,10-penta-isopropylpyrene, benzo[a]- and benzo[e]pyrene, and perylene; the yields, relative isomer distributions and the ^{19}F NMR data are shown.

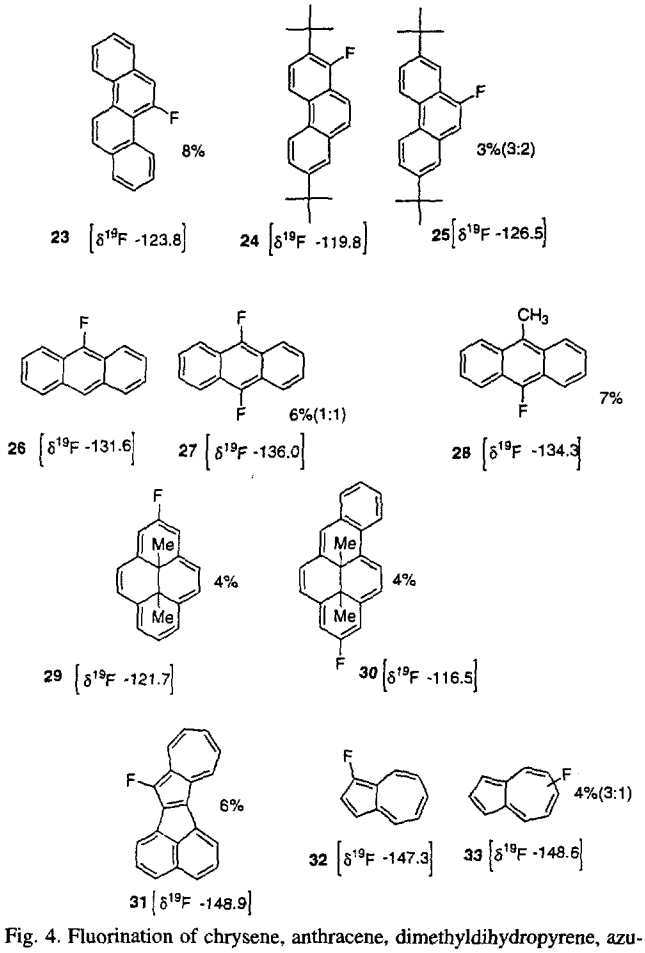

Fig. 4. Fluorination of chrysene, anthracene, dimethyldihydropyrene, azuleno[1,2-a] acenaphthylene and azulene; the yields, relative isomer distributions, and the ^{19}F NMR data are shown.

by-product (Fig. 3). Essentially the same products were obtained by independent XeF_2 fluorination of benzo[a]-pyrene [18]. Whereas the XeF_2 fluorination of benzo[a]-pyrene and formation of **16** as the only fluorinated product had been reported [18], PAH dimer formation had not been mentioned.

7-Methylbenzo[a]pyrene: Fluorination occurred at the C-6 to give **17** (Fig. 3); a PAH dimer was also formed. The products were separated by preparative HPLC and analyzed by NMR (see also Section 3). The same products were formed via independent XeF_2 fluorination of 7-methyl-benzo[a]pyrene. The ^{19}F NMR spectrum of **17** shows a deshielding of ca. 10 ppm relative to **16** due to a *peri*-effect by the 7-methyl group.

Benzo[e]pyrene: Fluorination of benzo[e]pyrene gave three isomeric monofluoro compounds (**18**, **19**, **20**; m/z 270) in 19% overall yield, which could not be separated; the relative assignments are, therefore, tentative (Fig. 3). In addition, GC-MS analysis indicated the presence of minor amounts of the difluoro compound (m/z 288).

Perylene: Fluorination gave two regioisomers **21**, **22** (m/z 270) in a 2:1 ratio with a total yield of 27% (Fig. 3). Moreover, GC-MS analysis indicated the presence of trace amounts of difluoro-perylene (m/z 288).

Chrysene (Fig. 4): gave 6-fluorochrysene **23** [41], as the sole fluorinated product but only in 8% yield.

2,7-Di-*tert*-butylphenanthrene: Fluorination occurred at C-1 and at the *meso* position to give **24** and **25** (m/z 308) in 3:2 ratio albeit in poor yield (Fig. 4).

Anthracene and 9-methylanthracene: Fluorination of anthracene occurred at the *meso* positions to give the 9-fluoro-**26** [42,20–22] and the 9,10-difluoroanthracene **27** [39] in 1:1 ratio (Fig. 4). The difluoro derivative **27** could be separated from the monofluoro compound. The observed ^{19}F NMR value δ −136.0 (**27**) differs somewhat from the literature value (δ −131.9 [40]). 9-Methylanthracene fluorinated selectively at C-10 to give **28**.

Trans-15,16-Dimethyldihydropyrene DMDHP and its benzo-fused Derivative: Fluorination of DMDHP and its benzannelated derivative both occurred at C-2 (→**29** and **30**), but the yields were quite low and extensive decomposition occurred (Fig. 4). The observed site of fluorination in DMDHP is the same as that in other aromatic substitution reactions of this [14] annulene [43–48].

Azuleno[1,2-a]acenaphthylene and Azulene: In line with its bromination [49], azuleno-acenaphthylene fluorinated selectively at C-7 to give **31**, albeit in only 6% yield. For azulene, a mixture of two isomers **32**, **33** were obtained in 3:1 ratio in low yield (Fig. 4).

Benzo[ghi]perylene, coronene, 1,6,7,12-tetramethylperylene: Whereas benzo[ghi]perylene and corenone were both fluorinated, the yields and isomer ratios could not be determined due to separation difficulties and solubility problems. Attempted fluorination of 1,6,7,12-tetramethylperylene [50], led to extensive side reactions, forming a complex mixture which could not be identified.

Other (attempted) Fluorinations: 1-methylnaphthalene, 2-methylphenanthrene, 4*H*-cylopenta[def]phenanthrene and triphenylene did not react with **1** and intact PAHs were recovered after 3 days reflux. In an effort to force the fluorination of 1-methylnaphthalene, it was reacted with **1** in decahydronaphthalene as solvent under reflux. Again, no fluorination could be induced.

2.2. Summary

The fluorination ability of **1** has been tested towards a wide variety of polynuclear aromatics; over 20 compounds have been examined with the yields varying from 27% to 3% depending on the aromatic substrate. The utilized simple one-pot approach appears promising for large PAHs, allowing direct access to their fluorinated analogs for further elaboration. The observed reduced yields with increasing PAH ionization potentials and the formation of PAH dimers in several cases point to a radical cation mechanism for these 'transfer-fluorinations'. The observed regioselectivity for benzo[a]-anthracene resembles that found in anodic oxidation. Decreased fluorination yields and increased formation of tar by increasing the **1**:PAH ratio is also indicative of an oxidative fluorination pathway. Formation of fluorodealkylation side-product with crowded alkyl(cycloalkyl)pyrenes is noteworthy.

3. Experimental

The PAHs and annulenes used in this survey study were either available in our laboratory from previous work or were commercially available compounds of highest purities. Compound **1** was prepared according to Refs. [33–35].

Caution: Although we have not experienced any problems, polynitro compounds are potentially dangerous. Adequate shielding and safety measures should be applied while handling them.

A number of the mono- and difluorinated products formed in this work are known compounds (e.g., **3, 4, 5, 6, 7, 8, 9, 23, 26, 27**) [17–23,38–41]; samples of **3, 4, 8** and **9** were received from Prof. P.E. Hansen (Roskilde). An authentic sample of **16** was prepared by XeF_2 fluorination [18] and purified by preparative HPLC; its 7-methyl derivative **17** was similarly prepared by XeF_2 fluorination and HPLC purification. In addition to ^{19}F NMR ($CFCl_3$ was used as internal standard and $CDCl_3$ as lock), **16** and **17** were both fully characterized by ^{1}H, ^{13}C, H/H COSY and C/H HETCOR analysis. NMR spectra were recorded on a GE-GN300 MHz instrument using a 5 mm fluorine probe and a 5 mm C/H switchable probe.

3.1. General procedure for the fluorinations

The PAH (0.1 mmol) and the fluorinating reagent **1** (0.11 mmol) were added to dichloroethane (20 ml) in a 50 ml three-necked flask. The reaction mixture was refluxed for 3 days with stirring during which time, 2,4-dinitroimidazole slowly precipitated out of the reaction during the fluorination. After cooling, the reaction mixture was poured into water and extracted with chloroform. After removal of the solvent under reduced pressure, the residue was analyzed by GC (OV101 capillary column) to determine the yields via the internal standard method. The products were separated by column chromatography using hexane as an eluent (or by HPLC) and the cuts were monitored by GC. In general, the fluorinated products had shorter retention times than the starting materials in LC. The retention times for the fluorinated products was also shorter in the GC than those of the starting materials, except for 1,2,3,6,7,8-hexahydropyrene, 4,5,9,10-tetrahydropyrene, and dimethyldihydropyrene.

The identity of the fluorinated products was based on a comparison of the GC and NMR data for each LC fraction or with authentic compounds (see also Section 2). In the case of low yielding reactions leading to inseparable mixtures, GC-MS analyses confirmed the formation of the fluoro compounds (see Section 2) and ^{19}F NMR data (Figs. 2–4) were used to estimate the isomer distribution. In the case of 1,3,6,8-tetra-isopropylpyrene, the byproduct had a longer R_f in the LC than that of the product, but its GC retention time was far shorter than both the product and the intact PAH, suggesting fluorodealkylation, confirmed by GC-MS (m/z 346). The R_f values decreased in the LC with increasing number of alkyl substituents, thus increasing the difficulty in the separation of the fluorinated products from the PAHs.

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