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^{1}H, ^{13}C, ^{15}N, and ^{19}F random coil NMR shifts of trifluoromethyl-bearing amino acids

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ABSTRACT

Fluorine-labeling is a powerful technique in biomolecular NMR, yet its application is often hindered by a lack of standardized reference data for non-natural residues. In this work, we report the multinuclear random coil chemical shifts (^{1}H, ^{13}C, ^{15}N, and ^{19}F) for three commercially available, trifluoromethyl-bearing amino acids: trifluoroaminobutyric acid (TfAbu), trifluoronorvaline (TfNva), and 2-trifluoromethyltryptophan ((2-Tfm)Trp). These residues exhibit steric profiles comparable to the canonical amino acids Val, Leu, and Trp, respectively, making them suitable reporters for on-site ^{19}F-labeling of proteins. Random coil data was recorded using a well-established hexapeptide model (Gly-Gly-X-Ala-Gly-Gly). The obtained chemical shifts are highly consistent with the dataset established by Wishart et al. (1995), thereby providing the foundational framework for NMR-based structure elucidation of complex, fluorine-labeled biomolecules.

1. Introduction

Fluorine-labeling is as a powerful technique in biomolecular NMR spectroscopy [1]. The ^{19}F isotope is characterized by its 100% natural abundance and an expansive chemical shift range that is highly sensitive to changes in its local chemical environment. Moreover, the absence of endogenous fluorine in almost all organisms effectively eliminates background interference. The use of trifluoromethyl ($-CF_3$) groups further amplifies signal-to-noise ratio and narrows line-width due to the threefold increase of active nuclei and the rapid rotation of the methyl group, which averages chemical shift anisotropy [2]. Fluorinated amino acids, made accessible by scalable organic synthesis [3,4] allow for the direct incorporation of ^{19}F probes into peptides and proteins. Consequently, trifluoromethyl-bearing amino acids are rapidly emerging as reporters for in-cell ^{19}F NMR and magnetic resonance imaging (MRI) [5–7].

To this end, the analysis of secondary chemical shifts presents a straightforward method for estimating protein secondary structure and has become indispensable for NMR-based protein structure elucidation. This approach operates on the principle that the secondary shift ($\Delta\delta$), calculated as the difference between the observed shift and its corresponding random coil value (δ_{rc}), serves as a sensitive reporter of the local chemical environment. Specifically, consistent positive or negative deviations in protein backbone (H, C, Hα, and Cα) shifts are characteristic of α-helix or β-sheet conformations [8,9]. Side-chain shift deviations can provide critical insights into tertiary effects, such as aromatic ring current influences, salt-bridge formation, or hydrogen-bonding interactions. The utility of this method relies on highly accurate reference data. Wishart, Sykes and coworkers were the first to provide a complete, internally consistent set of ^{1}H, ^{13}C, and ^{15}N random coil chemical shifts for the 20 canonical amino acids [10]. Their experimental approach, utilizing a synthetic hexapeptide model with the sequence Gly-Gly-X-Y-Gly-Gly (where X is the amino acid of interest and Y is the nearest neighbor to X), has since been widely adapted to obtain reference data for non-natural amino acids and we have employed it in previous studies for norleucine [11] and the 4-fluoro, 5-fluoro, 6-fluoro, and 7-fluoro regioisomers of tryptophan (Table 1) [12].

Here we report the random coil chemical shifts of three non-natural, trifluoromethyl-bearing amino acids, namely trifluoroaminobutyric acid (TfAbu), trifluoronorvaline (TfNva), and 2-trifluoromethyltryptophan ((2-Tfm)Trp). We discuss intrinsic spectroscopic properties, including nearest-neighbor effects of these residues on sequential Ala and report the UV absorbance of (2-Tfm)Trp.

2. Experimental

Materials. TentaGel XV RAM resin, Fmoc-(2-Tfm)Trp-OH, and

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Fmoc-TfAbu-OH were purchased from Iris Biotech GmbH (Markredwitz, Germany). Fmoc-TfNva-OH was purchased from abcr GmbH (Karlsruhe, Germany). Ac-Gly-OH, Fmoc-Ala-OH, and Fmoc-Gly-OH were purchased from Carbolution Chemicals GmbH (St. Ingbert, Germany). N,N'-Diisopropylcarbodiimide (DIC), Oxyma, other reagents, and solvents of at least p.a. grade quality were acquired from Carl Roth GmbH + Co. KG (Karlsruhe, Germany), Merck KGaA (Darmstadt, Germany), and Fisher Scientific GmbH (Schwerte, Germany).

Peptide synthesis. Peptides were prepared by solid-phase peptide synthesis using a Liberty Blue 2.0 (CEM Corporation, Matthews, NC, USA) automated synthesizer at 0.05–0.10 mmol scale on TentaGel XV Rink Amide resin (0.24 mmol/g, 100 μm mesh). Fmoc-amino acids (5 equiv) were coupled using DIC/Oxyma (5 equiv) at 90 °C for 2 min and Fmoc was removed using 10% piperidine in DMF at 90 °C for 1 min. Fluorinated Fmoc-amino acids (1.5 equiv) were coupled at 75 °C for 10 min. The resin was cleaved in TFA/TIS/water (95:2.5:2.5) for 3 h at room temperature and the crude peptide was precipitated in cold diethyl ether. Crude peptides were purified by preparative reversed-phase HPLC over a Kinetex C18 column (Phenomenex, Torrance, CA, USA). Peptide identity was confirmed by electrospray ionization time-of-flight mass spectrometry (ESI-TOF MS) and NMR spectroscopy. MS data were obtained as monoisotopic masses for Ac-Gly-Gly-TfAbu-Ala-Gly-Gly-NH_2: *m/z* calcd for $[M + H]^+$ $C_{17}H_{27}F_3N_7O_7$ 498.1919, found 498.1947; Ac-Gly-Gly-TfNva-Ala-Gly-Gly-NH_2: *m/z* calcd for $[M + H]^+$ $C_{18}H_{29}F_3N_7O_7$ 512.2075, found 512.2136; Ac-Gly-Gly-(2-Tfm)Trp-Ala-Gly-Gly-NH_2: *m/z* calcd for $[M + H]^+$ $C_{25}H_{32}F_3N_8O_7$ 613.2341, found 613.2375 (Fig. S1 in the Supporting Information).

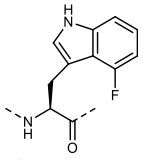
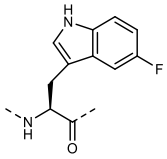
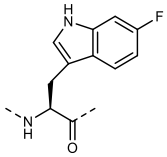
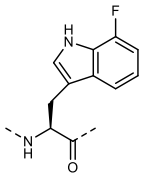
Nuclear magnetic resonance (NMR) spectroscopy. Unless stated otherwise NMR spectra were recorded on a 700 MHz AVANCE III spectrometer (Bruker BioSpin, Billerica, MA, USA) equipped with a TCI ($^1H/^{13}C/^{15}N$) cryo probe at 298 K. Spectra were recorded using a solution of ~3 mM random coil peptide, 50 mM sodium phosphate (pH 5.8), 1.0 M urea, and 0.1 mM 2,2-dimethyl-2-silapentane-5-sulfonic acid (DSS) in 10% D_2O/90% H_2O in a 5 mm Boroeco NMR tube (Deutero GmbH, Kastellaun, Germany). Proton spectra were directly referenced to DSS. Indirect chemical shift referencing was applied to ^{13}C and ^{15}N using the IUPAC-IUB recommended chemical shift referencing ratios Ξ of 0.251449530 (^{13}C-^1H), and 0.101329118 (^{15}N-^1H), respectively [13, 14].

Fluorine spectra were recorded on a 400 MHz JNM-ECA400II spectrometer (Jeol, Ltd., Akishima, Tokyo, Japan) at 298 K from –225.06 to 25.26 ppm using 64 scans and a relaxation delay of 5.0 s. Fluorine spectra were directly referenced to trifluoroacetic acid (TFA, –75.25 ppm) [15] remaining from peptide synthesis.

NMR data were processed with Bruker TopSpin 4.3.0 and MestReNova 14.2, respectively. 2D NMR data were assigned using NMRFAM-Sparky 1.3 (Sparky 3.12). ^1H, ^{13}C, and ^{15}N NMR spectra are shown in the Supporting Information (Fig. S2, S3, and S4). The chemical shift assignments are made publicly available on the BioMagResBank (BMRB) [16] under the accession codes 53609, 53611, and 53612.

UV spectroscopy. The molar absorption coefficient $\varepsilon_{280\ nm}$ of (2-Tfm)Trp was determined using a previously reported procedure [17]. Ac-Gly-Gly-(2-Tfm)Trp-Ala-Gly-Gly-NH_2 (10.0 mg) was precisely weighed into a graduated flask and dissolved in Milli-Q water (10.0 mL). From this stock solution dilute samples with a UV absorbance in the range of 0.1–1.0 A.U. were prepared and UV absorption spectra were recorded on a CARY 50 BIO UV–VIS spectrophotometer (Varian Medical Systems, Steinhausen, Switzerland) in a Hellma QS quartz cuvette with 10.00 mm optical path length against Milli-Q water as a blank. The absorbance values at 280 nm were plotted against the sample concentration and $\varepsilon_{280\ nm}$ was determined by linear regression. This procedure was repeated three times to obtain $\varepsilon_{280\ nm} = 5470 \pm 130$ L mol^{-1} cm^{-1}

Table 1
Previously reported random coil chemical shifts of fluorine-substituted tryptophans [12].

Residue	H	C	N	Hα	Cα	Hβ	Cβ	Others
(4-F)Trp	8.26	176.1	121.2	4.69	58.1	3.34, 3.28	30.9	H-1 10.43, H-2 7.20, H-5 6.84, H-6 7.16, H-7 7.29, C-2 127.9, C-3 109.7, C-4 119.1, C-5 107.0, C-6 125.3, C-7 111.0, C-8 141.9, C-9 118.1, N-1 132.4, F-4 –124.2
								
(5-F)Trp	8.28	176.1	121.6	4.64	57.5	3.24, 3.21	29.7	H-1 10.29, H-2 7.31, H-4 7.31, H-6 7.02, H-7 7.44, C-2 129.3, C-3 111.6, C-4 105.7, C-5 119.1, C-6 112.9, C-7 115.5, C-8 135.5, C-9 129.9, N-1 129.8, F-5 –124.7
								
(6-F)Trp	8.29	176.1	121.7	4.65	57.5	3.24, 3.24	29.6	H-1 10.26, H-2 7.24, H-4 7.57, H-5 6.96, H-7 7.21, C-2 127.8, C-3 111.6, C-4 121.9, C-5 110.7, C-6 119.1, C-7 100.6, C-8 138.8, C-9 126.3, N-1 130.7, F-6 –121.5
								
(7-F)Trp	8.30	176.1	121.6	4.66	57.4	3.26, 3.26	29.7	H-1 10.65, H-2 7.30, H-4 7.42, H-5 7.10, H-6 6.99, C-2 128.3, C-3 112.4, C-4 117.0, C-5 122.6, C-6 109.5, C-7 119.1, C-8 127.1, C-9 133.6, N-1 124.1, F-7 –133.9
								

for (2-Tfm)Trp. The UV absorbance spectrum of (2-Tfm)Trp is shown in the Supporting Information (Fig. S6).

3. Results and discussion

Random coil chemical shifts of three non-natural, fluorinated amino acids were determined using the protocol established by Wishart et al. (Table 2) [10]. TfAbu, TfNva, and (2-Tfm)Trp were incorporated into hexapeptides with the sequence Ac-Gly-Gly-X-Ala-Gly-Gly-NH_2 (where X is the amino acids of interest) as shown in Fig. 1. At the time of writing, all three fluorine-substituted amino acids and their respective Fmoc-building blocks were commercially available. Peptides were synthesized by Fmoc-chemistry on solid support and purified by reversed-phase HPLC. The molar absorption coefficient of (2-Tfm)Trp at 280 nm ($\varepsilon_{280\ nm} = 5470 \pm 130$ L mol^{-1} cm^{-1}) was determined from the UV absorbance of the respective random coil hexapeptide for photometric concentration measurement [17].

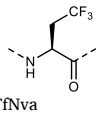
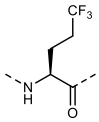
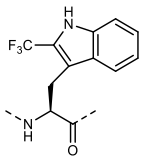
To ensure our NMR data was consistent with previous chemical shift sets and to prevent peptide aggregation, NMR spectra were recorded at 298 K in acidic (pH 5.8), low-concentration solutions containing 1 M urea denaturant, with direct referencing to 2,2-dimethyl-2-silapentane-5-sulfonic acid (DSS). Chemical shift assignments were based on 2D NMR experiments (TOCSY, COSY, ^{1}H–^{13}C HSQC, and ^{1}H-^{15}N HSQC). Quaternary trifluoromethyl carbons as well as their respective adjacent carbon atoms exhibited characteristic heteronuclear carbon–fluorine coupling patterns (^{13}C NMR spectra are shown in Fig. S3 in the Supporting Information). The extracted *J*-coupling constants (Table 3) revealed that the aliphatic residues exhibited larger one-bond coupling (TfAbu: $^{1}J_{CF\gamma} = 277$ Hz, TfNva: $^{1}J_{CF\delta} = 277$ Hz, respectively) than the aromatic Trp analogue ($^{1}J_{CF\text{-}2M} = 269$ Hz). Conversely, a higher ^{2}J constant was observed for (2-Tfm)Trp, consistent with the enhanced transmission of nuclear spin information across the conjugated indole π-system. Beyond these positions, no long-range carbon–fluorine couplings ($^{3}J_{CF}$ or further) were resolved. ^{19}F NMR data were referenced directly to trifluoroacetic acid using the reference value for H_2O/D_2O established by Rosenau et al. ($\delta_{TFA} = -75.25$ ppm) [15]. Fluorine-spectra (shown in Fig. 1) revealed three-bond heteronuclear coupling for the aliphatic amino acids (TfAbu: $^{3}J_{H\beta-F\gamma} = 10.5$ Hz, TfNva: $^{3}J_{H\gamma-F\delta} = 10.8$ Hz), while no proton–fluorine coupling was observed for (2-Tfm)Trp.

To explore fluorine-specific effects on intrinsic NMR spectroscopic properties, we compared the observed random coil values of the trifluoromethyl-bearing residues to those of their respective native analogues. While random coil chemical shifts for Trp have been established by Wishart et al. [10] to the best of our knowledge, no comparable reference data for α-aminobutyric acid and norvaline (Nva) were available in the literature at the time of this study (with the exception of δ_{rc}(Abu Hα) = 4.20 ppm) [18]. Subtraction of the native Trp random coil values revealed significant chemical shift deviations for (2-Tfm)Trp H-1 (+1.20 ppm), C-2 (−1.6 ppm), C-3 (+2.6 ppm), C-5 (+2.4 ppm), C-6 (+2.8 ppm), and Cβ (−1.2 ppm) which arguably result from the altered electronic environment of the trifluoromethylated indole moiety (all chemical shift deviations are shown in Table S1, and Fig. S5 in the Supporting Information). Cross-comparison of the random coil shifts observed for TfAbu and TfNva exemplified the effects of fluorination depending on side-chain length. Proton signals appear increasingly downfield-shifted the closer their proximity to the $-CF_3$ group (for TfAbu: Hβ +0.75 ppm, Hα +0.31 ppm, H +0.24 ppm vs. TfNva), while ^{13}C and ^{15}N resonances are displaced upfield. Nearest-neighbor effects of the trifluoromethyl-bearing amino acids were investigated, by comparing the chemical shifts of the sequential Ala4 residue (Table S2 in the Supporting Information). Notably, when preceded by (2-Tfm)Trp, Ala4 proton shifts were universally displaced upfield by about −0.1 ppm. As Wishart et al. point out, this is caused by time-averaged ring current effects from the proximal aromatic side chain [10]. When preceded by TfAbu or TfNva, Ala4 H was displaced downfield by +0.3 ppm while other nuclei showed no discernible deviations.

4. Conclusion

We report the random coil chemical shifts of all active nuclei (^{1}H, ^{13}C, ^{15}N, and ^{19}F) for three trifluoromethyl-bearing amino acids (TfAbu, TfNva, and (2-Tfm)Trp). Chemical shifts were directly referenced to DSS (^{1}H) or TFA (^{19}F) [15] or indirectly referenced using the IUPAC recommended chemical shift referencing ratios (^{13}C, and ^{15}N) [13,14]. We obtained the molar absorption coefficient $\varepsilon_{280\ nm}$ of aromatic (2-Tfm)Trp to facilitate spectrophotometric quantification of proteins modified with this non-natural amino acid. By maintaining consistent experimental conditions, our data directly extends the set of random coil chemical shifts established by Wishart et al. [10]. As synthetic and *in vivo* techniques continue to expand the repertoire of non-canonical amino acids in protein engineering, the extension of NMR databases ensures that secondary shift analysis remains a robust diagnostic tool for the rapid structure elucidation of complex, increasingly modified biomolecular systems.

Table 2
Random coil chemical shifts of TfAbu, TfNva, and (2-Tfm)Trp.

Residue	H	C	N	Hα	Cα	Hβ	Cβ	Others
TfAbu 	8.61	174.0	117.8	4.73	51.0	2.86, 2.69	36.8	Cγ 128.5, Fγ −63.9 (t)
TfNva 	8.37	174.5	119.4	4.42	55.3	2.11, 1.96	26.4	Hγ 2.28, 2.28, Cγ 32.1, Cδ 129.7, Fδ −65.9 (t)
(2-Tfm)Trp 	8.27	175.5	120.7	4.64	57.5	3.46, 3.35	28.4	H-1 11.30, H-4 7.72, H-5 7.25, H-6 7.39, H-7 7.54, C-2 125.8, C-3 113.8, C-4 122.5, C-5 123.4, C-6 127.6, C-7 115.2, C-8 138.4, C-9 129.5, C-2M 124.7, N-1 127.7, F-2M −57.7 (s)

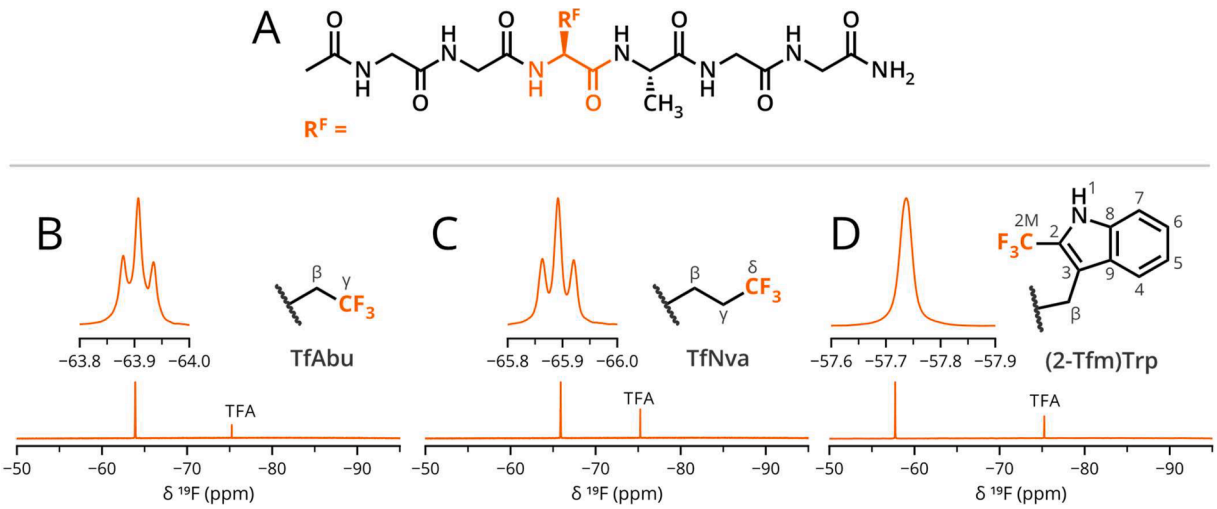


Fig. 1. Random coil ^{19}F NMR spectra of three trifluoromethyl-bearing amino acids. Fluorine spectra were recorded in 10% D_2O/90% H_2O at 298 K and were directly referenced to trifluoroacetic acid (TFA, −75.25 ppm) [15] **A** Peptide design adapted from Wishart et al. [10]. Fluorinated amino acids (orange) were incorporated into hexapeptides with the sequence Ac-Gly-Gly-X-Ala-Gly-Gly-NH_2. **B** Trifluoroaminobutyric acid (TfAbu). **C** Trifluorononorvaline (TfNva). **D** 2-Trifluoromethyltryptophan ((2-Tfm)Trp).

Table 3
Carbon–fluorine and fluorine–proton coupling constants observed for trifluoromethyl-bearing amino acid. Carbon spectra were recorded on a 700 MHz spectrometer and fluorine spectra were recorded on a 400 MHz spectrometer, respectively.

Residue	$^1J_{CF}$ (Hz)	$^2J_{CF}$ (Hz)	$^3J_{HF}$ (Hz)
TfAbu	277	29	10.5
TfNva	277	29	10.8
(2-Tfm)Trp	269	37	

The commercially available amino acids TfAbu, TfNva, and (2-Tfm)Trp are suitable probes for ^{19}F protein NMR owing to the favorable magnetic properties of the –CF_3 group and their steric profiles, comparable to canonical Val, Leu, and Trp, respectively. The multinuclear random coil dataset established in this work provides a detailed characterization of their intrinsic spectroscopic properties, thereby laying the foundation for their application as ^{19}F-labels in biomolecular structural studies.

CRediT authorship contribution statement

David Reiter: Writing – original draft, Investigation, Data curation, Conceptualization. **Selin Türk:** Investigation, Data curation. **Mario Schubert:** Validation, Supervision, Resources, Investigation, Data curation, Conceptualization. **Beate Koksch:** Supervision, Resources, Funding acquisition, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.jfluchem.2026.110567.

Data availability

The data that support the findings of this study are openly available in the Biological Magnetic Resonance Data Bank at https://bmrb.io, reference numbers 53609, 53611, and 53612.

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