Spectroscopic separation of mixed organic fluorine compounds by 2D ^{19}F–^{19}F TOCSY

—To extract 1D ^{19}F NMR spectrum of each compound—

Product used：Nuclear Magnetic Resonance (NMR)

Perfluoroalkyl compounds are often found in research fields such as pharmaceuticals and agrochemicals. There are increasing demands for 1D ^{19}F NMR, which is a powerful tool to characterize these compounds. Sensitivity of ^{19}F is the second highest after ^{1}H, which facilitates to obtain a ^{19}F NMR spectrum. Furthermore, the observation range of ^{19}F chemical shifts in fluorocarbons is very large and signals show good dispersion. However, ^{19}F NMR analysis of a mixture of these compounds are generally very complicated. This is because similarity of the chemical properties of each compound often occurs overlaps of the signals. For the same reason, purification by using chromatographic techniques is difficult. Therefore, it's convenient if a 1D ^{19}F NMR spectrum of each compound is directly obtained from a mixture.

2D TOCSY is known as a method which enables to obtain a 1D NMR spectrum from a mixture. To measure a ^{19}F–^{19}F TOCSY [1] with high field NMR equipment, a radio frequency (RF) covering wide spectrum width of ^{19}F is necessary. However, it's difficult to use such a RF, so ^{19}F TOCSY has not been generally used. Recently, to address the wide ^{19}F chemical shift range, 2D ^{19}F TOCSY with BURBOP spin lock (Fig. 1) was reported [2]. Here, this application note explains a method to extract a 1D ^{19}F spectrum of each compound from a mixture by using 2D ^{19}F TOCSY with BURBOP spin lock.

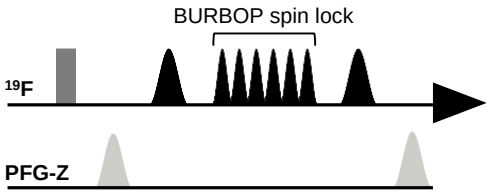

Fig. 1. ^{19}F TOCSY pulse sequence with BURBOP spin lock.

Spectroscopic separation of a mixture by 2D ^{19}F TOCSY

In 2D TOCSY, when measured with a sufficiently enough long mixing time, magnetization transfer occurs in all coupled spins. Therefore, a 1D ^{19}F NMR spectrum of each compound can be extracted. As an example, Figure. 2 shows a 2D ^{19}F TOCSY of two organic fluorine compounds. Two sliced data were made at -121.8 ppm (red dotted line) and -126.8 ppm (blue dotted line), respectively. Figure. 3 shows (a) the 1D ^{19}F spectrum of the mixture, (b) the sliced data at -121.8 ppm, and (c) at -126.8 ppm. Each sliced data is equivalent to the 1D ^{19}F spectrum of each compound. As you can see, each 1D ^{19}F spectrum is successfully extracted from 2D ^{19}F TOCSY.

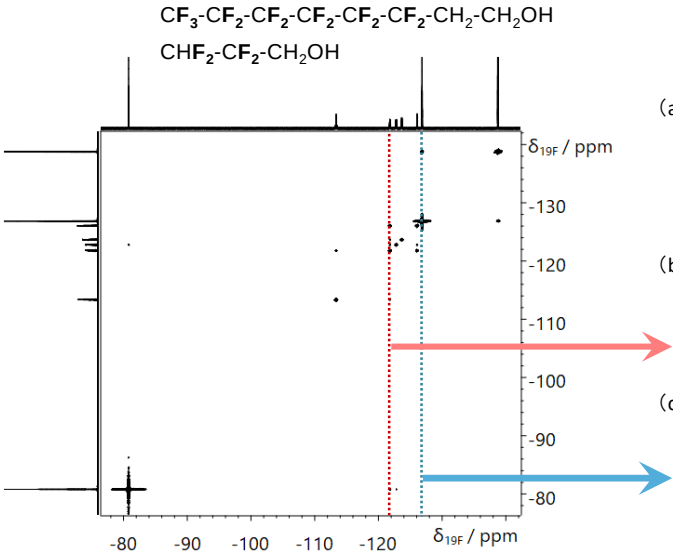

Fig. 2. ^{19}F TOCSY spectrum of a mixture of two organic fluorine compounds.

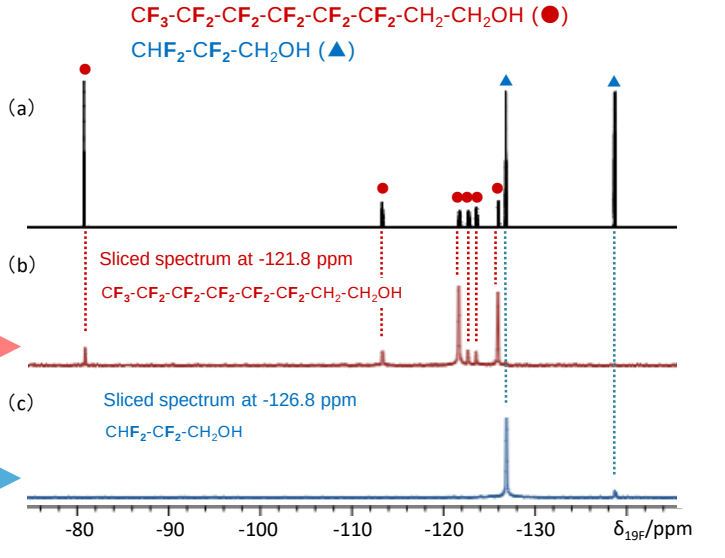

Fig. 3. (a) 1D ^{19}F NMR spectrum of the mixture, (b) sliced spectrum at -121.8 ppm, and (c) sliced spectrum at -126.8 ppm.

Equipment: JNM-ECZL400S (Delta NMR software V6.3), ROYALPROBE™ HFX, Pulse sequence: 19f_tocsy_burbop_abs_pfg.jxp,
Sample: 2,2,3,3-tetrafluoro-1-propanol and 3,3,4,4,5,5,6,6,7,7,8,8,8-tridecafluoro-1-octanol in $CDCl_3$

[1] *Anal. Chem.*, **65**, (1993) 752-758, [2] *J. Magn. Reson.*, **285**, (2018) 143-147.

JEOL Ltd. 3-1-2 Musashino Akishima Tokyo 196-8558 Japan Sales Division Tel. +81-3-6262-3560 Fax. +81-3-6262-3577
www.jeol.com ISO 9001 • ISO 14001 Certified
▼ Local office
• AUSTRALIA & NEW ZEALAND • BELGIUM • BRAZIL • CANADA • CHINA • EGYPT • FRANCE • GERMANY • GREAT BRITAIN & IRELAND • ITALY • KOREA • MALAYSIA • MEXICO • RUSSIA • SCANDINAVIA • SINGAPORE • TAIWAN • THE NETHERLANDS • USA