Catalytic Oxidative Benzylation of Amines Enabled by Direct Ionic Decarboxylation

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We report a method for the decarboxylative alkylation of amines via the direct generation of a benzylic nucleophiles.

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Catalytic Oxidative Benzylation of Amines Enabled by Direct Ionic Decarboxylation

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Abstract Decarboxylative C(sp³)–N substitution reactions are an emerging class of amination protocols that avoid the preparation of sensitive alkyl (pseudo)halides, improving process efficiency and scope. However, stoichiometric activation of the carboxylic acid with high molecular weight reagents is necessary to induce a radical decarboxylation required for coupling. We demonstrate an approach in which aryl acetic acids undergo ionic decarboxylation to enable the direct benzylation of nitrogen nucleophiles via oxidative Cu-catalysis. The reaction proceeds from native carboxylic acids at room temperature, is compatible with protic, electrophilic, and other potentially complicating functionality, allowing the direct functionalization of complex amines.

The strategic formation of C(sp³)–N bonds is of paramount importance for the synthesis of functional molecules.¹ The benzylic amine fragment is a particularly significant unit found in a diverse array of pharmaceuticals and clinical candidates, including Imatinib, Abemaciclib, Lacosamide, Clopidogrel, among many others.² Substitution between a nitrogen nucleophile and a benzylic (pseudo)halide and reductive amination of carbonyl compounds are standard approaches to benzylic amines. These textbook reactions are widely used in drug discovery, comprising nearly 10% of all transformations reported in synthetic medicinal chemistry journals in 2008,³ with increase in use reported in 2014.⁴ Alkyl electrophiles used to generate benzylic amines can be tedious to prepare and exhibit modest stability. Selective nucleophilic substitution of these species can be complicated by the presence of other protic or electrophilic groups, limiting utility in the synthesis of complex targets. Thus, the development mechanistically distinct synthetic methods that use alternative N-benzylating reagents stand to directly impact the preparation of complex nitrogen-containing molecules.^{6–8}

Decarboxylative C(sp³)–N coupling strategies using readily available, bench-stable carboxylic acids can increase versatility in complex amine synthesis the synthesis by circumventing the need for electrophilic N-alkylating reagents. Decarboxylative N-alkylation processes that proceed via C–carboxyl homolysis to form carbon-centered radicals have been reported (**Fig 1a, path I**).⁹ These indirect methods require the stoichiometric modification of the carboxylic acid with high molecular weight activators like N-hydroxyphthalimide (NHPI)^{9b,9c} or hypervalent iodine reagents (**A*** in **Fig 1**),^{9a, 9d, 9e} hampering process step economy and overall reaction efficiency. Decarboxylation must be paired with a selective amination step in order to avoid non-productive quenching of the radical intermediate. In these cases, intermolecular coupling is restricted to poorly nucleophilic substrates (imide,^{9b} anilines,^{9c, 9e} sulfonamides,^{9d, 9e} N-heterocycles^{9e}). A mechanistically distinct, but unknown, amination approach involves the direct ionic decarboxylation and trapping of native carboxylic acids, which would bypass stoichiometric carboxylate activation and streamline alkyl amine synthesis (**Fig. 1a, path II**). Control over the generation of the nucleophilic intermediate in tandem with an appropriate catalyst/oxidant system would allow for selective amination in the presence of other reactive functionality. We describe such an approach herein using electron-poor aryl acetic acids as selective N-benzylating reagents (**Fig. 1b**).¹⁰ This method leverages ionic decarboxylation to enable the controlled generation of benzylic nucleophiles, where Cu serve to both mediate the redox coupling steps and prevent undesirable substrate protodecarboxylation in the presence of protic nucleophiles by slowing the release of reactive intermediate.¹¹ The mechanistic steps of the oxidative coupling likely parallel those established for the Cu-catalyzed coupling of amines and aryl boronic acids (Chan–Evans–Lam reaction) to engender broad functional group compatibility.^{12, 13}

Reaction development studies culminated in conditions that allowed the decarboxylative amination of aryl acetate **1a** with piperidine to give **2a** in 92% yield using 30 mol% CuI and MnO₂ as the oxidant (**Fig. 2a**). The free acid could be used with the addition of mild base (94% yield). The cost-effective nature of MnO₂ (~\$0.10/g) made

Figure 1. (A) Decarboxylative amination of carboxylic acids. (B) Mechanistic hypothesis for the direct oxidative amine benzylation by the decarboxylation of aryl acetic acids.

it an attractive oxidant, reactions conducted under air gave reasonable product yield (50%), while the use of pure O_2 gave dimerization product and effectively no cross-coupling. A number of Cu-based catalysts afforded **2a** in good yield (CuI , $\text{Cu}(\text{OAc})_2$, $\text{Cu}(\text{OTf})_2$). Other metal salts including Pd, Ni, Co, or Fe-species were inferior to CuI but afforded some benzylated product (see the SI for additional data). To rapidly assess functional group compatibility, a tolerance screen was conducted under the standard conditions.¹⁴ High product yields without consumption of additive was observed in the presence of alkyl chlorides, aldehydes, alcohols, amides, sulfonamides, olefins, and basic N-heterocycles (**Fig 2a**). Chemoselectivity for coupling at the benzylic carboxylate unit was observed in the presence of aryl or primary alkyl carboxylic acids (terminal alkynes were not tolerated, see the SI).

The generality of the benzylation process was explored with other classes of nucleophiles (**Fig 2b**). Secondary or primary alkyl amines and NaN_3 were alkylated in good yields (**2a–2c**, 57–98%) under the standard conditions, with ~10% product generated in the absence of catalytic CuI . The amine scope of compliments radical decarboxylative aminations that require less-nucleophilic substrates.⁹ Sulfonates, thiolates, and malonate underwent benzylation in good to excellent yields (**2e–2g**, 61–90%), but did not require Cu catalysts. Unlike amine-based nucleophiles which can be coupled to a range of electron-poor aryl acetates (Fig 4), sulfur and malonate nucleophiles provide synthetically useful yields only with nitro-activated partners. Aniline and phenol were resistant to benzylation (<2% yield).

Decarboxylation is a key step in the reaction pathway; Cu enhances the stability of the aryl acetate substrates, preventing undesirable protodecarboxylation. Under the conditions in **Fig 2a**, the remaining aryl acetate mass

Figure 2. (A) Reaction development and functional group compatibility. (B) Nucleophile classes for oxidative benzylation (C) Impact of protic nucleophiles and additives on protodecarboxylation (D) Direct coupling of alkene-tethered aryl acetate. See SI for full details.

balance is alkylated arene **3a** (~5%). Acid **1a** is stable in DMF, however the addition of protic nucleophiles (piperidine or EtOH), led to rapid generation of **3a**, as does activated MnO₂ which is partially hydrated (**Fig 2c**). Catalytic amounts of Cu salts suppress protodecarboxylation in the presence of both protic nucleophiles (EtOH) and MnO₂, in agreement with past studies that showed Cu(aryl acetate)₂ species are slower to decarboxylate than the corresponding alkali metal salts.^{10b} The nature of the decarboxylation step was explored by subjecting an olefin-tethered aryl acetate substrate (**1t**) to the standard conditions (**Fig 2c**). Only direct amine coupling product was observed (**2t**, 64% yield) along with proto-decarboxylated material (**3t** 27% yield). Radical cyclization products were not observed, likely excluding the intermediacy of a benzylic radical formed by homolytic decarboxylation of a carboxyl radical or oxidation of a benzylic anion.¹⁵ Substrate **1a** was also found to add to aldehyde electrophiles to generate homobenzylic alcohols in the absence of catalyst or oxidant to further support the generation of a nucleophilic benzyl intermediate (>80% yield with PhCHO, see SI for details).¹⁶

Mechanistic controls further support a pathway in which decarboxylation occurs prior to C–N bond formation via a Chan–Evans–Lam-type cycle (**Fig 3**). Both ester substrate **4a** and amino carboxylate **4b** do not generate product under the standard conditions and are recovered in high yield. These observations rule out a dienolate carbonyl α -amination pathway (see the SI for details).^{17,10a} Consistent with a direct oxidative amination mechanism,

A Decarboxylation is Essential for Amination and Occurs as the First Step

B Evidence for a Chan–Lam Type Redox Pathway

Figure 3. (A) Viability of alternative substrates or intermediates (B) Product yields under varying Cu(II) loadings. Ar = 2- or 4-NO₂C₆H₄, R = H, Me, or *n*-Pr.

stoichiometric deprotonation of the ethyl-substituted nitroarene **4c** with KH under standard conditions provided the amination product in low, but observable yield (~10%, **Fig 3a**). Decomposition of the putative benzylic nucleophile intermediate is observed under these conditions, highlighting the importance of a controlled release of organometallic species to achieve efficient cross-coupling. Various loadings of Cu(OAc)₂ deliver product with less than 0.5 turnover number (TON) in the absence of an external oxidant. (**Fig. 3b**). This is consistent with two equivalents of Cu(II) being required to form one equivalent of product, analogous to the Chan-Evans-Lam mechanism.¹³ Under catalytic conditions MnO₂ or O₂ likely serve to re-oxidize the Cu(I) generated in the bond forming process.¹⁸

With an understanding of mechanism and functional group compatibility, the scope of the reaction was explored (**Fig. 4**). Cyclic or acyclic secondary amines derivatives provided the corresponding benzylic amines in good to excellent yields. Selective N-benylation was achieved in the presence of other heteroatom-containing functionality including other protected or unprotected amines (**5d–5f**, **5h**), alcohols (**5g**), 1°/2° amides (**5i**, **5j**), and sulfur-containing heterocycles (**5c**, **5k**). N-benylation in the presence of an electrophilic aldehyde (**5m**) was achieved, complementing reductive amination protocols. Primary amines could be benzylated, albeit in lowered yields (**5r–5u**, 52–57%) Bis-alkylation was not observed in these cases which facilitated easy isolation. Marketed amine drug molecules bearing sensitive functionality including aryl fluorides and chlorides, functionalized N- and S-heterocycles and a tetrasubstituted olefin, were alkylated in good yields (Paroxetine, Debenzylidonepezil, Desloratadine, Crizotinib Fasudil, and Duloxetine; **5v–5aa**).

Figure 4. Scope and application. Unless noted, yields are of isolated material; [a] with 25 mol% Zn(OAc)₂, [b] Cu(OAc)₂ instead of CuI, [c] CuBr₂ instead of CuI, [d] with 1 equiv. Zn(OAc)₂, [e] 1:1 DCE/DMF. See SI for details and less successful example.

The scope of aryl acetic acid partner was investigated using piperidine. A range of electron-deficient aryl acetate derivatives proved suitable partners, including 4- and 2-nitro (**6a**, **6f**), 4- and 2-cyano (**6b**, **6g**), 4-sulfonyl (**6c**), 4-acetyl (**6d**), 4-benzoyl (**6e**) and 4-pyridyl (**6h**) derivatives. Substituents on the aryl acetic acid partner were well tolerated, including halogens (**6j**, **6m**, **6u**) or trifluoromethyl (**6q**) groups and electron-donating methoxy (**6i**, **6k**, **6o**) or dimethylamino (**6l**) groups. Successful benzylation was achieved using aryl acetic acids bearing other sensitive functionality such as free or silyl-protected alcohols (**6r**, **6s**), a terminal olefin (**2t**), and a ketal (**6u**). Under the standard conditions, this protocol was extended to achieve decarboxylative azidation using NaN₃. Azidation of functionalized 2- and 4-nitrophenyl acetic acids could be achieved in moderate to excellent yields (**2c**, **7a–7h**). The process is currently restricted to electronically activated benzyl partners; however, a series of diverse targets could be obtained in good overall yields by simple nitro-group functionalizations, including conversions to anilines (**8**, **9**). Further modifications by aromatic substitution to access products of arene deamination (**10**), iodination (**11**), hydroxylation (**12**) or Heck cross-coupling (**13**) was achieved in overall yields of 52–76%.

The formal synthesis of a family of histone deacetylase/bromodomain and extra-terminal protein (HDAC/BET) inhibitors¹⁹ was accomplished using the decarboxylative azidation protocol, showcasing the method's potential utility in complex molecules synthesis. Decarboxylative azidation of **14a** to form **14b** was conducted on gram scale in 92% yield. Target **14b** contains differentiated amino-precursor units that can be chemoselectively reduced and acylated to yield the key amino tetrahydroquinoline pharmacophore. Azide reduction and carbamate installation, acetal deprotection, and one-pot nitro-group reduction/diastereoselective reductive amination afforded the advanced intermediate of DUAL946 (**14f**) in 61% yield over 4 steps.

In summary, chemoselective N-benylation of amines can be achieved directly from native carboxylic acids in the presence of protic or electrophilic groups via oxidative Cu-catalysis. The Cu-controlled ionic decarboxylation pathway enables the slow liberation of a benzylic nucleophile to allow for efficient oxidative trapping with basic alkyl amines, including those found in complex pharmaceuticals. The scope of reaction partners compliments radical decarboxylative amination approaches. The benzylic amine products can be expediently diversified, showcasing this method's potential in complex molecule synthesis. Efforts are underway to apply this concept in the development of related chemoselective ionic decarboxylative cross-coupling reactions

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Contents

I.	General Considerations	S-2
II.	Selected Important Molecules Containing the Benzylic Amine Motif	S-2
III.	Selected Optimization Data	S-3
IV.	Functional Group Compatibility Screens	S-4
V.	Mechanistic Experiments	S-6
VI.	Effect of Zn(OAc)₂ Additives	S-7
VII.	Unsuccessful Examples	S-7
VIII.	Cu-Catalyzed Decarboxylative Amination of Phenylacetic Acid Derivatives	S-8
IX.	Synthesis of Tetrahydroquinoline Core for DUAL946	S-45
X.	References	S-49
XI.	¹H and ¹³C Spectra	S-50

I. General Considerations

Unless noted, all reactions were conducted under inert atmosphere employing standard Schlenk technique or by the use of a N₂-filled glovebox. All glassware was oven-dried prior to use. Flash chromatography was performed as described by Still and co-workers¹ (SiliaFlash P60, 40-63µm, 60A silica gel, Silicycle) or by automated flash chromatography (Ultra SNAP silica cartridges, Biotage). Analytical thin-layer chromatography was performed using glass plates pre-coated with silica (SiliaPlate G TLC - Glass-Backed, 250µm, Silicycle). TLC plates were visualized by UV light and/or staining with aqueous basic potassium permanganate. Unless otherwise noted, all reagents were obtained from commercial vendors and used as supplied. Manganese dioxide (activated, ~85%, Sigma-Aldrich). Potassium aryl acetate salts **1h** and **1l** (starting materials of **6h** and **6l**) were synthesized from the corresponding aryl acetic esters as described by Liu and co-workers.² Other potassium aryl acetate salts were synthesized from the corresponding aryl acetic acids as described by Liu and co-workers.³

II. Selected Important Molecules Containing the Benzylic Amine Motif

Figure S1. Selected important molecules containing the benzylic amine motif

III. Selected Optimization Data

Effect of Key Reaction Parameters

entry	deviation from standard conditions	conv. 1a	2a (%)
1	none	>99	92
2	Air instead of MnO ₂	>99	50
3	O ₂ instead of MnO ₂	80	<2
4	K ₂ S ₂ O ₈ instead of MnO ₂	39	<2
5	H ₂ O ₂ instead of MnO ₂	73	5
6	TBHP instead of MnO ₂	81	7
7	Ag ₂ CO ₃ instead of MnO ₂	78	36
8	DMSO instead of DMF	>99	83
9	DMA instead of DMF	>99	61
10	NMP instead of DMF ^a	>99	64
11	Cu(OAc) ₂ instead of CuI	>99	91
12	Cu(OTf) ₂ instead of CuI	65	60
13	Zn(OAc) ₂ instead of CuI	>99	15
14	Pd(OAc) ₂ instead of CuI	>99	40
15	CoI ₂ instead of CuI	63	10
16	NiI ₂ instead of CuI	76	11
17	Fe(OAc) ₂ instead of CuI	>99	11
18	acid instead of carboxylate ^b	92	76
19	acid instead of carboxylate ^c	>99	94

^a 48h; ^b 50 °C; ^c addition of 1.0 equiv. K₂CO₃, 30h.

Figure S2. Effect of reaction parameters on decarboxylative amination

IV. Functional Group Compatibility Screens

entry	Additive	yield (%)	Additive remaining (%)	entry	Additive	yield (%)	Additive remaining (%)
1	none	88 ✓	n.d.	10 ^c		69 -	>99 ✓
2		86 ✓	>99 ✓	11 ^e		80 ✓	>99 ✓
3 ^a		89 ✓	>99 ✓	12		87 ✓	>99 ✓
4		89 ✓	94 ✓	13		76 ✓	>99 ✓
5		88 ✓	>99 ✓	14		58 -	0 ✗
6 ^c		67 -	>99 ✓	15		78 ✓	64 -
7 ^d		90 ✓	>99 ✓	16		77 ✓	76 ✓
8		91 ✓	97 ✓	17		89 ✓	>99 ✓
9		84 ✓	>99 ✓	18		85 ✓	>99 ✓

Reactions performed on 0.10 mmol scale according to the general procedure (glovebox), with an additional 1.0 equivalent of additive. Yields determined by calibrated NMR using durene as internal standard. General assessment of product yield and additive recovery based on : 100–75% (good ✓), 75–50% (mediocre –), 50–0% (poor ✗). n.d. not determined.

^a 28h; ^b 48h; ^c 5d; ^d an additional of 1.0 equiv of K₂CO₃, 41h; ^e an additional of 1.0 equiv of K₂CO₃, 3d.

Figure S3. Functional group compatibility screen using piperidine

entry	Additive	yield (%)	Additive remaining (%)	entry	Additive	yield (%)	Additive remaining (%)
1	none	95 ✓	n.d.	7		95 ✓	93 ✓
2		75 ✓	89 ✓	8		89 ✓	93 ✓
3		97 ✓	91 ✓	9		92 ✓	93 ✓
4		90 ✓	>99 ✓	10		66 -	<5 ✗
5		84 ✓	93 ✓	11		59 -	56 -
6		99 ✓	>99 ✓	12		86 ✓	77 ✓

Reactions performed on 0.10 mmol scale according to the general procedure (glovebox), with an additional 1.0 equivalent of additive. Yields determined by calibrated NMR using durene as internal standard. General assessment of product yield and additive recovery based on : 100–75% (good ✓), 75–50% (mediocre -), 50–0% (poor ✗). n.d. not determined.

Figure S4. Functional group compatibility screen using NaN_3

V. Mechanistic Experiments

Potential ionic decarboxylative amination pathways

Figure S5. Potential pathways for the decarboxylative amination

Figure S6. Experiment of trapping the benzylic anion with benzaldehyde

Under N₂ atmosphere, **1a** undergoes rapid decarboxylative alkylation of benzaldehyde.

Procedure: In a glovebox filled with N₂, potassium aryl acetate (52.3 mg, 0.2 mmol, 1.0 equiv.), trimethoxybenzene (0.4 equiv.) and anhydrous DMF (1.0 mL) were added sequentially to a 1-dram vial charged with a stir bar. A homogeneous light red solution forms upon stirring. The vial was sealed with a PTFE-lined cap, removed from the glovebox. The solution is sparged ~1 minute with either a balloon of N₂, then the balloon is installed as to maintain a headspace of N₂. Benzaldehyde (27 μ L, 0.26 mmol, 1.3 equiv.) is added by volume. The light red homogeneous solution under N₂ immediately becomes light yellow upon addition of benzaldehyde. The reactions were monitored by ¹H NMR using trimethoxybenzene as internal standard. The reaction under N₂ was by diluted with EtOAc (50 mL) and quenched with 1 M HCl (3 mL), and washed with H₂O (8 x 20 mL), then brine (20 mL). The organic layer was dried with Na₂SO₄, and concentrated in vacuo. Isolated in 74% (3:1 dr) yield as a pale yellow oil after purification by silica column chromatography (10:1 to 4:1 hexane:EtOAc).

¹H NMR (CDCl₃, 500 MHz) δ [*major diastereomer*] 7.65 (m, 1H), 7.49 (m, 1H), 7.41 (m, 1H), 7.38 – 7.21 (m, 6H), 4.97 (m, 1H), 3.65 (m, 1H), 2.01 (d, *J* = 3.6 Hz, 1H), 1.83 – 1.76 (m, 2H), 1.13 – 1.00 (m, 2H), 0.79 (t, *J* = 7.4 Hz, 3H); [*minor diastereomer*] 7.67 (m, 1H), 7.61 – 7.58 (m, 2H), 7.38 – 7.21 (m, 6H), 4.70 (m, 1H), 3.65 (m, 1H), 2.21 (d, *J* = 4.7 Hz, 1H), 1.63 (m, 1H), 1.44 (m, 1H), 1.13 – 1.00 (m, 2H), 0.74 (t, *J* = 7.4 Hz, 3H);

¹³C NMR (CDCl₃, 125 MHz) δ [*major diastereomer*] 151.6, 142.3, 136.0, 131.7, 129.9, 128.2, 127.5, 127.1, 126.2, 123.8, 77.2, 46.2, 30.9, 20.7, 14.0;

HRMS (ESI): calcd for C₁₇H₁₉NNaO₃ [*M*+Na]⁺: 308.1257. Found 308.1257.

VI. Effect of Zn(OAc)₂ Additives

Figure S7. Effect of Zn(OAc)₂ additives

VII. Unsuccessful Examples

Figure S8. Unsuccessful substrates under standard conditions

VIII. Cu-Catalyzed Decarboxylative Amination of Phenylacetic acid Derivatives

General Procedure A: In a glovebox filled with N₂, Cu salts (0.3 equiv.), MnO₂ (5.0 equiv), potassium aryl acetate (1.0 equiv.), Zn(OAc)₂ (0 – 1.0 equiv.), amine or NaN₃ (1.5 - 3.0 equiv) and anhydrous DMF (0.2 M) or DMF/DCE (1:1, 0.2 M) were added sequentially to a 1 dram vial charged with a stir bar. The vial was sealed with a PTFE-lined cap, removed from the glovebox and stirred at the corresponding temperature. ¹H NMR analysis of small aliquots (~5 µL) was used to follow reactions to completion, afterwhich the mixture was diluted in ethyl acetate (80 mL) and washed sequentially with saturated NaHCO₃ (20 mL x 2) and brine (20 mL). The organic layer was dried over Na₂SO₄, concentrated *in vacuo* and purified by flash column chromatography.

General Procedure B: In a glovebox filled with N₂, Cu salts (0.3 equiv.), MnO₂ (5.0 equiv), aryl acetic acid (1.0 equiv.), K₂CO₃ (0 - 1 equiv.), amine or NaN₃ (1.5 - 3.0 equiv) and anhydrous DMF (0.2 M) or DMF/DCE (1:1, 0.2 M) were added sequentially to a 1 dram vial charged with a stir bar. The vial was sealed with a PTFE-lined cap, removed from the glovebox and stirred at the corresponding temperature. ¹H NMR analysis of small aliquots (~5 µL) was used to follow reactions to completion, afterwhich the mixture was diluted in ethyl acetate (80 mL) and washed sequentially with saturated NaHCO₃ (20 mL x 2) and brine (20 mL). The organic layer was dried over Na₂SO₄, concentrated *in vacuo* and purified by flash column chromatography.

2a Prepared according to the General Procedure A from the corresponding amine (1.5 mmol, 3.0 equiv.), potassium aryl acetate (0.5 mmol, 1.0 equiv.), CuI (0.15 mmol, 0.3 equiv.), and MnO₂ (2.5 mmol, 5.0 equiv) in 2.5 mL DMF, 30 °C, 18h. Isolated in 85% yield after purification by silica gel chromatography (4:1 hexane:EtOAc) as a light yellow oil.

Prepared according to the General Procedure B from the corresponding amine (1.5 mmol, 3.0 equiv.), aryl acetic acid (0.5 mmol, 1.0 equiv.), K₂CO₃ (0.5 mmol, 1.0 equiv.), CuI (0.15 mmol, 0.3 equiv.), and MnO₂ (2.5 mmol, 5.0 equiv) in 2.5 mL DMF, 30 °C, 30h. 94% yield determined by ¹H NMR using durene as internal standard.

¹H NMR (CDCl₃, 700 MHz) δ 7.61 (d, *J* = 8.2 Hz, 1H), 7.54-7.48 (m, 2H), 7.33 (t, *J* = 8.2 Hz, 1H), 3.95 (q, *J* = 4.7 Hz, 1H), 2.35-2.27 (m, 4H), 1.88-1.84 (m, 1H), 1.74-1.68 (m, 1H), 1.46 (brs, 4H), 1.35-1.33 (m, 2H), 1.27-1.17 (m, 2H), 0.89 (t, *J* = 7.5 Hz, 3H);

¹³C NMR (CDCl₃, 176 MHz) δ 151.6, 136.5, 131.2, 128.9, 127.3, 123.5, 62.9, 51.0, 33.0, 26.2, 24.6, 19.5, 14.3;

HRMS (EI): calcd for C₁₅H₂₂N₂O₂ [M]⁺: 262.1681. Found 262.1681.

2b Prepared according to the General Procedure A from the corresponding amine (1.5 mmol, 3.0 equiv.), potassium aryl acetate (0.5 mmol, 1.0 equiv.), CuI (0.15 mmol, 0.3 equiv.), Zn(OAc)₂ (0.125 mmol, 0.25 equiv.), and MnO₂ (2.5 mmol, 5.0 equiv) in 2.5 mL DMF, rt, 48h. Isolated in 57% yield after purification by silica gel chromatography (4:1 hexane:EtOAc) as a yellow oil.

¹H NMR (CDCl₃, 700 MHz) δ 7.72 (td, *J* = 8.3 Hz, 1.4 Hz, 2H), 7.56 (td, *J* = 8.2 Hz, 1.4 Hz, 1H), 7.34 (td, *J* = 8.2 Hz, 1.4 Hz, 1H), 4.14 (t, *J* = 6.8 Hz, 1H), 2.45-2.41 (m, 1H), 2.33-2.29 (m, 1H), 1.69-1.64 (m, 2H), 1.42-1.25 (m, 7H), 0.89 (t, *J* = 7.5 Hz, 3H), 0.85 (t, *J* = 7.5 Hz, 3H);

¹³C NMR (CDCl₃, 176 MHz) δ 150.4, 139.8, 132.5, 128.6, 127.3, 123.6, 57.4, 47.6, 40.1, 32.4, 20.4, 19.6, 14.0, 13.9;

HRMS (ESI): calcd for C₁₄H₂₃N₂O₂ [M+H]⁺: 251.1754. Found 251.1750.

2c Prepared according to the General Procedure A from NaN₃ (1.0 mmol, 2.0 equiv.), the corresponding potassium aryl acetate (0.5 mmol, 1.0 equiv.), CuI (0.15 mmol, 0.3 equiv.), and MnO₂ (2.5 mmol, 5.0 equiv) in 2.5 mL DMF, 40 °C, 24h. Isolated in 87% yield after purification by silica gel chromatography (40:1 hexane:EtOAc) as a yellow oil.

¹H NMR (CDCl₃, 500 MHz) δ 7.94 (d, *J* = 8.3 Hz, 1H), 7.69-7.64 (m, 2H), 7.48-7.44 (m, 1H), 5.22 (q, *J* = 4.7 Hz, 1H), 1.83-1.70 (m, 2H), 1.57-1.39 (m, 2H), 0.96 (t, *J* = 7.4 Hz, 3H);

¹³C NMR (CDCl₃, 126 MHz) δ 148.2, 136.0, 133.6, 128.6, 128.5, 124.6, 60.9, 39.0, 19.6, 13.5;

HRMS (APPI): calcd for $C_{10}H_{13}N_2O_2$ $[M-N_2+H]^+$: 193.0972. Found 193.0970.

2e Prepared according to the General Procedure A from $PhSO_2Na$ (1.0 mmol, 2.0 equiv.), potassium aryl acetate (0.5 mmol, 1.0 equiv.), $Cu(OAc)_2$ (0.25 mmol, 0.5 equiv.), and MnO_2 (2.5 mmol, 5.0 equiv) in 2.5 mL DMF, 30 °C, 24h. 84% yield determined by 1H NMR using durene as internal standard. Isolated in 81% yield after purification by silica gel chromatography (10:1 hexane:EtOAc) as a yellow oil.

1H NMR ($CDCl_3$, 500 MHz) δ 7.85 (d, J = 7.8 Hz, 1H), 7.71-7.65 (m, 2H), 7.60-7.54 (m, 3H), 7.47-7.43 (m, 1H), 7.42-7.38 (m, 2H), 5.27 (dd, J = 11.2 Hz, 3.9 Hz, 1H), 2.43-2.36 (m, 1H), 2.26-2.18 (m, 1H), 1.38-1.24 (m, 2H), 0.91 (t, J = 7.3 Hz, 3H);

^{13}C NMR ($CDCl_3$, 126 MHz) δ 150.9, 137.3, 133.9, 133.0, 130.2, 129.5, 129.1, 128.6, 127.2, 124.8, 63.2, 30.0, 19.9, 13.7;

HRMS (ESI): calcd for $C_{16}H_{17}NO_4SNa$ $[M+Na]^+$: 342.0770. Found 342.0770.

2f Prepared according to the General Procedure A from sodium 4-methoxyphenylthiolate (1.0 mmol, 2.0 equiv.), potassium aryl acetate (0.5 mmol, 1.0 equiv.), and MnO_2 (2.5 mmol, 5.0 equiv) in 2.5 mL DMF, 30 °C, 24h. 61% yield determined by 1H NMR using durene as internal standard.

1H NMR ($CDCl_3$, 700 MHz) δ 7.65-7.61 (m, 2H), 7.53 (td, J = 7.6 Hz, 1.2 Hz, 1H), 7.29 (td, J = 7.9 Hz, 1.4 Hz, 1H), 7.11 (dt, J = 8.9 Hz, 3.2 Hz, 2H), 6.71 (dt, J = 8.9 Hz, 3.2 Hz, 2H), 4.79 (t, J = 7.5 Hz, 1H), 3.76 (s, 3H), 1.98-1.88 (m, 2H), 1.53-1.47 (m, 1H), 1.39-1.32 (m, 1H), 0.92 (t, J = 7.4 Hz, 3H);

^{13}C NMR ($CDCl_3$, 176 MHz) δ 159.9, 149.7, 137.7, 136.0, 132.5, 129.6, 127.3, 123.8, 123.7, 114.4, 55.3, 47.7, 37.8, 20.7, 13.8;

HRMS (ESI): calcd for $C_{17}H_{19}NO_3S$ $[M-H]^+$: 316.1013. Found 316.1007.

2g Prepared according to the General Procedure A from dimethyl malonate (1.0 mmol, 2.0 equiv.), potassium aryl acetate (0.5 mmol, 1.0 equiv.), CuI (0.15 mmol, 0.3 equiv.), BSA [N,O-Bis(trimethylsilyl)acetamide, 1.0 mmol, 2.0 equiv.], KOAc (0.1 mmol, 0.2 equiv.), and MnO₂ (2.5 mmol, 5.0 equiv) in 2.5 mL DMF, 30 °C, 72h. 90% yield determined by ¹H NMR using durene as internal standard. Isolated in 80% yield after purification by silica gel chromatography (10:1 hexane:EtOAc) as a yellow oil. (*Note: BSA, KOAc and DMF were pre-stirred for 15 min.*)

¹H NMR (CDCl₃, 500 MHz) δ 7.72 (dd, *J* = 8.2 Hz, 1.3 Hz, 1H), 7.53 (td, *J* = 7.8 Hz, 1.3 Hz, 1H), 7.39 (dd, *J* = 7.9 Hz, 1.2 Hz, 1H), 7.36-7.33 (m, 1H), 4.05 (td, *J* = 10.0 Hz, 1.4 Hz, 1H), 3.75 (s, 3H), 3.70 (d, *J* = 10.3 Hz, 1H), 3.49 (s, 3H), 1.79-1.68 (m, 2H), 1.36-1.25 (m, 1H), 1.13-1.03 (m, 1H), 0.91 (t, *J* = 7.3 Hz, 3H);

¹³C NMR (CDCl₃, 126 MHz) δ 168.2, 167.7, 151.5, 135.7, 132.4, 128.5, 127.6, 124.2, 58.3, 52.7, 52.6, 38.5, 35.8, 20.2, 14.1;

HRMS (ESI): calcd for C₁₅H₁₉NO₆Na [M+Na]⁺: 332.1105. Found 322.1102.

2t Prepared according to the General Procedure A from piperidine (1.5 mmol, 3.0 equiv.), the corresponding potassium aryl acetate (0.5 mmol, 1.0 equiv.), CuI (0.15 mmol, 0.3 equiv.), Zn(OAc)₂ (0.125 mmol, 0.25 equiv.), and MnO₂ (2.5 mmol, 5.0 equiv) in 2.5 mL DMF, 30 °C, 48h. Isolated in 83% yield after purification by silica gel chromatography (4:1 hexane:EtOAc) as a yellow oil.

¹H NMR (CDCl₃, 400 MHz) δ 7.63 (dd, *J* = 7.8 Hz, 1.0 Hz, 1H), 7.55-7.49 (m, 2H), 7.36-7.32 (m, 1H), 5.80-5.70 (m, 1H), 5.01-4.92 (m, 2H), 3.96 (q, *J* = 4.9 Hz, 1H), 2.37-2.24 (m, 4H), 2.09-2.01 (m, 2H), 1.95-1.86 (m, 1H), 1.80-1.70 (m, 1H), 1.49-1.42 (m, 4H), 1.39-1.21 (m, 4H);

¹³C NMR (CDCl₃, 100 MHz) δ 151.6, 138.5, 136.3, 131.3, 128.9, 127.3, 123.6, 114.8, 62.9, 51.0, 33.9, 30.1, 26.2, 25.5, 24.6;

HRMS (ESI): calcd for C₁₇H₂₅N₂O₂ [M+H]⁺: 289.1911. Found 289.1911.

5b Prepared according to the General Procedure A from the corresponding amine (1.5 mmol, 3.0 equiv.), potassium aryl acetate (0.5 mmol, 1.0 equiv.), CuI (0.15 mmol, 0.3 equiv.), and MnO₂ (2.5 mmol, 5.0 equiv) in 2.5 mL DMF, 30 °C, 18h. Isolated in 76% yield after purification by silica gel chromatography (4:1 hexane:EtOAc) as a light yellow oil.

¹H NMR (CDCl₃, 400 MHz) δ 7.64 (dd, *J* = 8.3 Hz, 1.4 Hz, 1H), 7.60 (dd, *J* = 8.3 Hz, 1.4 Hz, 1H), 7.53 (td, *J* = 7.5 Hz, 1.4 Hz, 1H), 7.36 (td, *J* = 7.6 Hz, 1.4 Hz, 1H), 3.90 (q, *J* = 4.4 Hz, 1H), 3.66-3.58 (m, 4H), 2.51-2.45 (m, 2H), 2.36-2.31 (m, 2H), 1.93-1.85 (m, 1H), 1.74-1.65 (m, 1H), 1.28-1.12 (m, 2H), 0.88 (t, *J* = 7.3 Hz, 3H);

¹³C NMR (CDCl₃, 176 MHz) δ 151.5, 135.9, 131.6, 129.2, 127.6, 123.6, 67.1, 63.1, 50.7, 33.2, 19.2, 14.2;

HRMS (EI): calcd for C₁₄H₂₀N₂O₃ [M]⁺: 264.1474. Found 264.1470.

5c Prepared according to the General Procedure A from the corresponding amine (1.5 mmol, 3.0 equiv.), potassium aryl acetate (0.5 mmol, 1.0 equiv.), CuI (0.15 mmol, 0.3 equiv.), Zn(OAc)₂ (0.125 mmol, 0.25 equiv.), and MnO₂ (2.5 mmol, 5.0 equiv) in 2.5 mL DMF, 30 °C, 18h. Isolated in 83% yield after purification by silica gel chromatography (8:1 hexane:EtOAc) as a yellow oil.

¹H NMR (CDCl₃, 400 MHz) δ 7.62 (dd, *J* = 8.0 Hz, 1.4 Hz, 1H), 7.49 (t, *J* = 8.0 Hz, 1H), 7.42 (d, *J* = 8.1 Hz, 1H), 7.36 (td, *J* = 8.0 Hz, 1.4 Hz, 1H), 4.14 (q, *J* = 4.8 Hz, 1H), 2.67 (t, *J* = 4.8 Hz, 4H), 2.56-2.46 (m, 4H), 1.87-1.71 (m, 2H), 1.33-1.25 (m, 2H), 0.94 (t, *J* = 7.4 Hz, 3H);

¹³C NMR (CDCl₃, 100 MHz) δ 151.6, 135.6, 131.2, 128.3, 127.7, 124.0, 62.8, 51.5, 30.7, 28.1, 20.0, 14.3;

HRMS (EI): calcd for C₁₄H₂₀N₂O₂S [M]⁺: 280.1245. Found 280.1247.

5d Prepared according to the General Procedure A from the corresponding amine (1.5 mmol, 3.0 equiv.), potassium aryl acetate (0.5 mmol, 1.0 equiv.), CuI (0.15 mmol, 0.3 equiv.), Zn(OAc)₂ (0.125 mmol, 0.25 equiv.), and MnO₂ (2.5 mmol, 5.0 equiv) in 2.5 mL DMF, 30 °C, 46h. Isolated in 84% yield after purification by silica gel chromatography (4:1 hexane:EtOAc) as a colourless solid.

¹H NMR (CDCl₃, 400 MHz) δ 7.62 (d, *J* = 8.0 Hz, 1H), 7.54-7.48 (m, 2H), 7.37-7.33 (m, 1H), 3.96 (q, *J* = 5.4 Hz, 1H), 3.32-3.27 (m, 4H), 2.42-2.37 (m, 2H), 2.29-2.27 (m, 2H), 1.89-1.81 (m, 1H), 1.73-1.64 (m, 1H), 1.41 (s, 9H), 1.26-1.12 (m, 2H), 0.87 (t, *J* = 7.4 Hz, 3H);

¹³C NMR (CDCl₃, 100 MHz) δ 154.6, 151.5, 135.8, 131.6, 128.9, 127.7, 123.7, 79.5, 62.5, 49.7, 44.0, 33.0, 28.4, 19.4, 14.2;

HRMS (ESI): calcd for C₁₉H₃₀N₃O₄ [M+H]⁺: 364.2231. Found 364.2232.

5e Prepared according to the General Procedure A from the corresponding amine (1.5 mmol, 3.0 equiv.), potassium aryl acetate (0.5 mmol, 1.0 equiv.), CuI (0.15 mmol, 0.3 equiv.), and MnO₂ (2.5 mmol, 5.0 equiv) in 2.5 mL DMF, 30 °C, 18h. Isolated in 69% yield after purification by silica gel chromatography (6:1 DCM:MeOH) as a light yellow oil.

¹H NMR (CDCl₃, 400 MHz) δ 7.61 (d, *J* = 8.3 Hz, 1H), 7.55-7.48 (m, 2H), 7.33 (td, *J* = 8.0 Hz, 1.8 Hz, 1H), 3.97 (q, *J* = 4.2 Hz, 1H), 2.48-2.28 (m, 8H), 2.23 (s, 3H), 1.91-1.82 (m, 1H), 1.75-1.66 (m, 1H), 1.29-1.12 (m, 2H), 0.88 (t, *J* = 7.3 Hz, 3H);

¹³C NMR (CDCl₃, 100 MHz) δ 151.5, 136.0, 131.5, 129.0, 127.5, 123.6, 62.4, 55.2, 49.6, 45.8, 33.0, 19.4, 14.2;

HRMS (EI): calcd for C₁₅H₂₂N₃O₂ [M-H]⁺: 276.1712. Found 276.1711.

5f Prepared according to the General Procedure A from the corresponding amine (1.5 mmol, 3.0 equiv.), potassium aryl acetate (0.5 mmol, 1.0 equiv.), CuI (0.15 mmol, 0.3 equiv.), and MnO₂ (2.5 mmol, 5.0 equiv) in 2.5 mL DMF, 30 °C, 18h. Isolated in 68% yield after purification by silica gel chromatography (1:3 DCM:MeOH) as a yellow oil.

¹H NMR (CDCl₃, 500 MHz) δ 7.62 (dd, *J* = 8.2 Hz, 1.2 Hz, 1H), 7.55 (dd, *J* = 8.2 Hz, 1.4 Hz, 1H), 7.50 (td, *J* = 7.6 Hz, 1.4 Hz, 1H), 7.33 (td, *J* = 8.2 Hz, 1.8 Hz, 1H), 3.93 (q, *J* = 4.7 Hz, 1H), 2.81-2.73 (m, 4H), 2.42-2.28 (m, 4H), 1.90-1.83 (m, 1H), 1.74-1.66 (m, 1H), 1.43 (s, 1H), 1.28-1.12 (m, 2H), 0.88 (t, *J* = 7.3 Hz, 3H);

¹³C NMR (CDCl₃, 126 MHz) δ 151.6, 136.1, 131.4, 129.1, 127.5, 123.6, 63.0, 51.3, 46.3, 32.9, 19.4, 14.3;

HRMS (ESI): calcd for C₁₄H₂₂N₃O₂ [M+H]⁺: 264.1707. Found 264.1705.

5g Prepared according to the General Procedure A from the corresponding amine (1.5 mmol, 3.0 equiv.), potassium aryl acetate (0.5 mmol, 1.0 equiv.), CuI (0.15 mmol, 0.3 equiv.), and MnO₂ (2.5 mmol, 5.0 equiv) in 2.5 mL DMF, 30 °C, 16h. Isolated in 81% yield after purification by silica gel chromatography (1:4 hexane:EtOAc) as a yellow oil.

¹H NMR (CDCl₃, 700 MHz) δ 7.62 (d, *J* = 7.6 Hz, 1H), 7.54- 7.50 (m, 2H), 7.35 (t, *J* = 7.6 Hz, 1H), 3.99 (q, *J* = 4.8 Hz, 1H), 3.61-3.57 (m, 1H), 2.74-2.72 (m, 1H), 2.63-2.61 (m, 1H), 2.17-2.08 (m, 2H), 1.88-1.69 (m, 4H), 1.49-1.43 (m, 2H), 1.33 (d, *J* = 5.0 Hz, 1H), 1.27-1.16 (m, 2H), 0.90 (t, *J* = 7.3 Hz, 3H);

¹³C NMR (CDCl₃, 176 MHz) δ 151.4, 136.3, 131.4, 128.8, 127.4, 123.6, 68.3, 62.2, 47.9, 47.0, 34.7, 34.6, 33.1, 19.5, 14.2;

HRMS (EI): calcd for C₁₅H₂₂N₂O₃ [M]⁺: 278.1631. Found 278.1627.

5h Prepared according to the General Procedure A from the corresponding amine (1.5 mmol, 3.0 equiv.), potassium aryl acetate (0.5 mmol, 1.0 equiv.), CuI (0.15 mmol, 0.3 equiv.), and MnO₂ (2.5 mmol, 5.0 equiv) in 2.5 mL DMF, 30 °C, 18h. Isolated in 68% yield after purification by silica gel chromatography (1:4 hexane:EtOAc) as a yellow oil.

¹H NMR (CDCl₃, 600 MHz) δ 7.96 (s, 1H), 7.65 (d, *J* = 7.8 Hz, 1H), 7.55-7.50 (m, 2H), 7.38 (t, *J* = 6.7 Hz, 1H), 4.07 (q, *J* = 4.6 Hz, 1H), 3.49-3.40 (m, 2H), 3.29-3.26 (m, 2H), 2.50-2.35 (m, 4H), 1.88-1.82 (m, 1H), 1.75-1.69 (m, 1H), 1.29-1.20 (m, 2H), 0.91 (t, *J* = 7.4 Hz, 3H);

¹³C NMR (CDCl₃, 176 MHz) δ 160.5, 151.4, 135.3, 131.6, 128.6, 127.9, 123.9, 62.3, 50.5, 48.7, 45.7, 40.1, 32.3, 19.6, 14.2;

HRMS (EI): calcd for C₁₅H₂₁N₃O₃ [M]⁺: 291.1583. Found 291.1587.

5i Prepared according to the General Procedure A from the corresponding amine (1.5 mmol, 3.0 equiv.), potassium aryl acetate (0.5 mmol, 1.0 equiv.), CuI (0.15 mmol, 0.3 equiv.), Zn(OAc)₂ (0.125 mmol, 0.25 equiv.), and MnO₂ (2.5 mmol, 5.0 equiv) in 2.5 mL DMF, 30 °C, 45h. Isolated in 80% yield after purification by silica gel chromatography (10:1 DCM:MeOH) as a light yellow oil.

¹H NMR (CDCl₃, 500 MHz) δ 7.60 (d, *J* = 8.0 Hz, 1H), 7.54-7.47 (m, 2H), 7.33 (td, *J* = 8.0 Hz, 1.0 Hz, 1H), 6.03 (brs, 1H), 5.64 (brs, 1H), 3.94 (q, *J* = 4.6 Hz, 1H), 2.87 (d, *J* = 12.0 Hz, 1H), 2.72 (d, *J* = 12.0 Hz, 1H), 2.06-1.94 (m, 3H), 1.85-1.53 (m, 6H), 1.24-1.12 (m, 2H), 0.86 (t, *J* = 7.4 Hz, 3H);

¹³C NMR (CDCl₃, 126 MHz) δ 177.9, 151.4, 136.3, 131.5, 128.9, 127.5, 123.6, 62.5, 50.5, 48.9, 42.9, 33.1, 29.2, 29.1, 19.4, 14.2;

HRMS (ESI): calcd for C₁₆H₂₄N₃O₃ [M+H]⁺: 306.1812. Found 306.1811.

5j Prepared according to the General Procedure B from the corresponding amine (1.5 mmol, 3.0 equiv.), aryl acetic acid (0.5 mmol, 1.0 equiv.), K₂CO₃ (0.50 mmol, 1.0 equiv.), CuI (0.15 mmol, 0.3 equiv.), and MnO₂ (2.5 mmol, 5.0 equiv) in 2.5 mL DMF, 30 °C, 45h. Isolated in 75% yield after purification by silica gel chromatography (15:1 DCM:MeOH) as a yellow oil.

¹H NMR (CDCl₃, 700 MHz) δ 7.68 (d, *J* = 7.7 Hz, 1H), 7.61 (d, *J* = 7.7 Hz, 1H), 7.57-7.55 (m, 1H), 7.41-7.38 (m, 1H), 6.05 (s, 1H), 4.02 (q, *J* = 4.2 Hz, 1H), 3.29-3.05 (m, 4H), 2.68-2.58 (m, 2H), 1.91-1.73 (m, 1H), 1.72-1.68 (m, 1H), 1.22-1.11 (m, 2H), 0.88 (t, *J* = 7.7 Hz, 3H);

¹³C NMR (CDCl₃, 176 MHz) δ 169.6, 151.2, 135.3, 132.1, 129.0, 128.1, 123.8, 62.0, 54.0, 46.9, 41.4, 33.7, 18.9, 14.2;

HRMS (ESI): calcd for C₁₄H₂₀N₃O₃ [M+H]⁺: 278.1499. Found 278.1502.

5k Prepared according to the General Procedure B from the corresponding amine (1.5 mmol, 3.0 equiv.), aryl acetic acid (0.5 mmol, 1.0 equiv.), K₂CO₃ (1.0 mmol, 2.0 equiv.), CuI (0.15 mmol, 0.3 equiv.), and MnO₂ (2.5 mmol, 5.0 equiv) in 2.5 mL DMF, 40 °C, 21h. Isolated in 62% yield after purification by silica gel chromatography (10:1 hexane:EtOAc) as a light yellow oil.

¹H NMR (CDCl₃, 500 MHz) δ 7.72-7.67 (m, 2H), 7.55 (t, *J* = 7.8 Hz, 1H), 7.37 (t, *J* = 7.8 Hz, 1H), 7.04 (d, *J* = 5.3 Hz, 1H), 6.69 (d, *J* = 5.3 Hz, 1H), 4.16 (q, *J* = 5.0 Hz, 1H), 3.74 (d, *J* = 14.4 Hz, 1H), 3.44 (d, *J* = 14.4 Hz, 1H), 2.83-2.66 (m, 4H), 2.03-1.97 (m, 1H), 1.84-1.79 (m, 1H), 1.30-1.15 (m, 2H), 0.91 (t, *J* = 7.4 Hz, 3H);

¹³C NMR (CDCl₃, 126 MHz) δ 151.4, 136.6, 133.9, 133.5, 131.9, 129.1, 127.7, 125.3, 123.6, 122.6, 62.1, 50.3, 48.2, 34.5, 25.9, 19.2, 14.3;

HRMS (ESI): calcd for C₁₇H₂₁N₂O₂S [M+H]⁺: 317.1318. Found 317.1317.

5l Prepared according to the General Procedure A from the corresponding amine (1.5 mmol, 3.0 equiv.), potassium aryl acetate (0.5 mmol, 1.0 equiv.), CuI (0.15 mmol, 0.3 equiv.), and MnO₂ (2.5 mmol, 5.0 equiv) in 2.5 mL DMF, 30 °C, 18h. Isolated in 73% yield after purification by silica gel chromatography (10:1 hexane:EtOAc) as a light yellow oil.

¹H NMR (CDCl₃, 700 MHz) δ 7.67-7.62 (m, 2H), 7.55 (dd, J = 7.5 Hz, 1.2 Hz, 1H), 7.38 (td, J = 8.0 Hz, 1.2 Hz, 1H), 7.26-7.23 (m, 2H), 6.89-6.83 (m, 3H), 4.02 (q, J = 4.5 Hz, 1H), 3.15-3.09 (m, 4H), 2.66-2.64 (m, 2H), 2.54-2.51 (m, 2H), 1.97-1.93 (m, 1H), 1.78-1.76 (m, 1H), 1.30-1.19 (m, 2H), 0.92 (t, J = 7.4 Hz, 3H);

¹³C NMR (CDCl₃, 176 MHz) δ 151.5, 151.3, 136.0, 131.6, 129.1, 129.0, 127.6, 123.6, 119.6, 116.0, 62.6, 50.0, 49.3, 33.4, 19.3, 14.3;

HRMS (ESI): calcd for C₂₀H₂₆N₃O₂ [M+H]⁺: 340.2020. Found 340.2017.

5m Prepared according to the General Procedure A from the corresponding amine (1.5 mmol, 3.0 equiv.), potassium aryl acetate (0.5 mmol, 1.0 equiv.), CuI (0.15 mmol, 0.3 equiv.), Zn(OAc)₂ (0.125 mmol, 0.25 equiv.), and MnO₂ (2.5 mmol, 5.0 equiv) in 2.5 mL DMF, 30 °C, 45h. Isolated in 53% yield after purification by silica gel chromatography (3:1 hexane:EtOAc) as a yellow oil.

¹H NMR (CDCl₃, 700 MHz) δ 9.76 (s, 1H), 7.71 (d, J = 8.6 Hz, 2H), 7.65 (dd, J = 8.0 Hz, 1.1 Hz, 1H), 7.59 (dd, J = 8.0 Hz, 1.2 Hz, 1H), 7.55 (td, J = 8.1 Hz, 1.2 Hz, 1H), 7.39 (td, J = 8.1 Hz, 1.2 Hz, 1H), 6.85 (d, J = 8.8 Hz, 2H), 4.01 (q, J = 4.4 Hz, 1H), 3.33-3.23 (m, 4H), 2.64-2.61 (m, 2H), 2.52-2.49 (m, 2H), 1.91-1.89 (m, 1H), 1.75-1.73 (m, 1H), 1.28-1.18 (m, 2H), 0.91 (t, J = 7.4 Hz, 3H);

¹³C NMR (CDCl₃, 176 MHz) δ 190.4, 155.0, 151.4, 135.7, 131.8, 131.7, 129.0, 127.8, 127.1, 123.7, 113.4, 62.5, 49.6, 47.3, 33.1, 19.3, 14.2;

HRMS (EI): calcd for C₂₁H₂₅N₃O₃ [M]⁺: 367.1896. Found 367.1905.

5n Prepared according to the General Procedure A from the corresponding amine (1.5 mmol, 3.0 equiv.), potassium aryl acetate (0.5 mmol, 1.0 equiv.), CuI (0.15 mmol, 0.3 equiv.), and MnO₂ (2.5 mmol, 5.0 equiv) in 2.5 mL DMF, 30 °C, 72h. Isolated in 48% yield after purification by silica gel chromatography (1:1 hexane:EtOAc) as a yellow solid.

¹H NMR (CDCl₃, 500 MHz) δ 7.64 (dd, J = 8.2 Hz, 1.4 Hz, 1H), 7.58 (d, J = 8.2 Hz, 1H), 7.52 (t, J = 8.6 Hz, 1H), 7.35 (td, J = 8.4 Hz, 1.8 Hz, 1H), 6.98 (d, J = 8.7 Hz, 2H), 6.20 (d, J = 8.5 Hz, 2H), 4.02 (q, J = 4.5 Hz, 1H), 3.53 (brs, 2H), 2.92 (d, J = 10.6 Hz, 1H), 2.81 (d, J = 10.6 Hz, 1H), 2.32-2.25 (m, 1H), 2.09-2.02 (m, 2H), 1.94-1.87 (m, 1H), 1.79-1.55 (m, 5H), 1.31-1.18 (m, 2H), 0.91 (t, J = 7.4 Hz, 3H);

¹³C NMR (CDCl₃, 126 MHz) δ 151.6, 144.5, 136.8, 136.4, 131.4, 129.0, 127.6, 127.4, 123.6, 115.2, 62.7, 51.7, 50.2, 42.0, 34.1, 33.8, 33.3, 19.6, 14.3;

HRMS (ESI): calcd for C₂₁H₂₈N₃O₂ [M+H]⁺: 354.2176. Found 354.2176.

5o Prepared according to the General Procedure A from the corresponding amine (1.5 mmol, 3.0 equiv.), potassium aryl acetate (0.5 mmol, 1.0 equiv.), CuI (0.15 mmol, 0.3 equiv.), and MnO₂ (2.5 mmol, 5.0 equiv) in 2.5 mL DMF, 30 °C, 18h. Isolated in 81% yield after purification by silica gel chromatography (2:1 hexane:EtOAc) as a yellow oil.

¹H NMR (CDCl₃, 400 MHz) δ 7.75 (d, J = 8.0 Hz, 1H), 7.62 (dd, J = 8.0 Hz, 1.2 Hz, 1H), 7.53 (td, J = 8.0 Hz, 1.2 Hz, 1H), 7.33 (td, J = 8.0 Hz, 1.2 Hz, 1H), 3.66 (dd, J = 9.3 Hz, 4.0 Hz, 1H), 2.57-2.54 (m, 2H), 2.35-2.31 (m, 2H), 1.97-1.88 (m, 1H), 1.77-1.67 (m, 5H), 1.25-1.01 (m, 2H), 0.82 (t, J = 7.3 Hz, 3H);

¹³C NMR (CDCl₃, 100 MHz) δ 150.8, 137.7, 132.0, 129.8, 127.3, 123.0, 63.5, 52.4, 38.0, 23.3, 18.6, 14.3;

HRMS (EI): calcd for C₁₄H₂₀N₂O₂ [M]⁺: 248.1525. Found 248.1525.

5p Prepared according to the General Procedure A from the corresponding amine (1.5 mmol, 3.0 equiv.), potassium aryl acetate (0.5 mmol, 1.0 equiv.), CuI (0.15 mmol, 0.3 equiv.), Zn(OAc)₂ (0.125 mmol, 0.25 equiv.), and MnO₂ (2.5 mmol, 5.0 equiv) in 2.5 mL DMF, rt, 48h. Isolated in 55% yield after purification by silica gel chromatography (4:1 hexane:EtOAc) as a light yellow oil.

¹H NMR (CDCl₃, 400 MHz) δ 7.59 (t, J = 7.6 Hz, 2H), 7.49 (t, J = 7.6 Hz, 1H), 7.33 (t, J = 7.6 Hz, 1H), 4.24 (q, J = 4.4 Hz, 1H), 2.56-2.41 (m, 4H), 1.89-1.81 (m, 1H), 1.73-1.63 (m, 1H), 1.29-1.11 (m, 2H), 0.92-0.88 (m, 9H);

¹³C NMR (CDCl₃, 100 MHz) δ 151.6, 137.7, 131.3, 129.0, 127.2, 123.4, 58.7, 43.3, 33.2, 19.6, 14.3, 12.4;

HRMS (EI): calcd for C₁₄H₂₂N₂O₂ [M]⁺: 250.1681. Found 250.1679.

5q Prepared according to the General Procedure A from the corresponding amine (1.5 mmol, 3.0 equiv.), potassium aryl acetate (0.5 mmol, 1.0 equiv.), CuI (0.15 mmol, 0.3 equiv.), and MnO₂ (2.5 mmol, 5.0 equiv) in 2.5 mL DMF, rt, 72h. Isolated in 54% yield after purification by silica gel chromatography (1:4 hexane:EtOAc) as a yellow oil.

¹H NMR (CDCl₃, 500 MHz) δ 7.64 (dd, J = 8.0 Hz, 1.2 Hz, 1H), 7.53 (td, J = 8.0 Hz, 1.3 Hz, 1H), 7.47 (dd, J = 8.0 Hz, 1.2 Hz, 1H), 7.38 (td, J = 8.0 Hz, 1.5 Hz, 1H), 4.19 (q, J = 5.0 Hz, 1H), 3.49 (t, J = 5.4 Hz, 2H), 2.53-2.41 (m, 2H), 2.34 (brs, 1H), 2.18 (s, 3H), 1.91-1.75 (m, 2H), 1.32-1.23 (m, 2H), 0.94 (t, J = 7.4 Hz, 3H);

¹³C NMR (CDCl₃, 126 MHz) δ 151.8, 135.3, 131.5, 128.4, 127.9, 123.8, 61.9, 58.5, 54.6, 37.6, 31.5, 19.9, 14.2;

HRMS (ESI): calcd for C₁₃H₂₁N₂O₃ [M+H]⁺: 253.1547. Found 253.1544.

5s Prepared according to the General Procedure A from the corresponding amine (1.5 mmol, 3.0 equiv.), potassium aryl acetate (0.5 mmol, 1.0 equiv.), CuI (0.15 mmol, 0.3 equiv.), Zn(OAc)₂ (0.125 mmol, 0.25 equiv.), and MnO₂ (2.5 mmol, 5.0 equiv) in 2.5 mL DMF, rt, 41h. Isolated in 52% yield after purification by silica gel chromatography (4:1 hexane:EtOAc) as a light yellow oil.

¹H NMR (CDCl₃, 400 MHz) δ 7.76-7.70 (m, 2H), 7.56 (td, J = 7.9 Hz, 1.3 Hz, 1H), 7.34 (td, J = 8.5 Hz, 1.5 Hz, 1H), 4.11 (t, J = 6.7 Hz, 1H), 2.27 (dd, J = 11.2 Hz, 6.4 Hz, 1H), 2.08 (dd, J = 11.2 Hz, 6.4 Hz, 1H), 1.71-1.58 (m, 3H), 1.45-1.24 (m, 3H), 0.90 (t, J = 7.4 Hz, 3H), 0.85 (t, J = 6.0 Hz, 6H);

¹³C NMR (CDCl₃, 100 MHz) δ 150.4, 140.0, 132.5, 128.7, 127.3, 123.6, 57.4, 55.9, 40.2, 28.6, 20.7, 19.6, 14.0;

HRMS (ESI): calcd for C₁₄H₂₃N₂O₂ [M+H]⁺: 251.1754. Found 251.1752.

5t Prepared according to the General Procedure A from the corresponding amine (1.5 mmol, 3.0 equiv.), potassium aryl acetate (0.5 mmol, 1.0 equiv.), CuI (0.15 mmol, 0.3 equiv.), and MnO₂ (2.5 mmol, 5.0 equiv) in 2.5 mL DMF, rt, 48h. Isolated in 54% yield after purification by silica gel chromatography (8:1 DCM:MeOH) as a light yellow oil.

¹H NMR (CDCl₃, 400 MHz) δ 7.74-7.68 (m, 2H), 7.61 (td, J = 7.4 Hz, 1.3 Hz, 1H), 7.38 (td, J = 7.4 Hz, 1.3 Hz, 1H), 4.20 (t, J = 6.7 Hz, 1H), 3.59-3.51 (m, 4H), 2.57-2.52 (m, 1H), 2.45-2.39 (m, 1H), 1.81-1.70 (m, 2H), 1.64-1.53 (m, 4H), 1.39-1.29 (m, 1H), 1.24-1.15 (m, 1H), 0.87 (t, J = 7.4 Hz, 3H);

¹³C NMR (CDCl₃, 100 MHz) δ 150.6, 137.4, 132.9, 128.5, 128.0, 123.8, 62.6, 57.2, 47.5, 39.1, 31.6, 27.8, 19.4, 13.9;

HRMS (ESI): calcd for C₁₄H₂₃N₂O₃ [M+H]⁺: 267.1703. Found 267.1705.

5u Prepared according to the General Procedure A from the corresponding amine (1.5 mmol, 3.0 equiv.), potassium aryl acetate (0.5 mmol, 1.0 equiv.), CuI (0.15 mmol, 0.3 equiv.), Zn(OAc)₂ (0.125 mmol, 0.25 equiv.), and MnO₂ (2.5 mmol, 5.0 equiv) in 2.5 mL DMF, rt, 70h. Isolated in 57% yield after purification by silica gel chromatography (4:1 hexane:EtOAc) as a light yellow oil.

¹H NMR (CDCl₃, 500 MHz) δ 7.71 (dd, J = 8.0 Hz, 1.4 Hz, 1H), 7.62 (dd, J = 8.0 Hz, 1.2 Hz, 1H), 7.52 (td, J = 7.6 Hz, 1.2 Hz, 1H), 7.34 (td, J = 8.5 Hz, 1.6 Hz, 1H), 7.29-7.26 (m, 2H), 7.21-7.18 (m, 1H), 7.15 (d, J = 7.7 Hz, 2H), 4.18 (t, J = 6.8 Hz, 1H), 2.76-2.71 (m, 3H), 2.65-2.60 (m, 1H), 1.66-1.59 (m, 2H), 1.41 (brs, 1H), 1.35-1.12 (m, 2H), 0.87 (t, J = 7.4 Hz, 3H);

¹³C NMR (CDCl₃, 126 MHz) δ 150.4, 139.9, 139.5, 132.6, 128.7, 128.6, 128.4, 127.3, 126.2, 123.6, 57.1, 48.8, 40.1, 36.4, 19.5, 13.9;

HRMS (ESI): calcd for C₁₈H₂₃N₂O₂ [M+H]⁺: 299.1754. Found 299.1751.

5v Prepared according to the General Procedure A from Paroxetine (0.3 mmol, 1.5 equiv.), potassium aryl acetate (0.2 mmol, 1.0 equiv.), CuI (0.06 mmol, 0.3 equiv.), Zn(OAc)₂ (0.05 mmol, 0.25 equiv.), and MnO₂ (1.0 mmol, 5.0 equiv) in 1.0 mL DMF, 30 °C, 50h. 63% yield (1:1 dr) determined by ¹H NMR using Bn₂O as internal standard.

Prepared according to the General Procedure A from Paroxetine (1.5 mmol, 3.0 equiv.), potassium aryl acetate (0.5 mmol, 1.0 equiv.), CuI (0.15 mmol, 0.3 equiv.), Zn(OAc)₂ (0.125 mmol, 0.25 equiv.), and MnO₂ (2.5 mmol, 5.0 equiv) in 2.5 mL DMF, 30 °C, 52h. Isolated in 74% yield (1:1 dr) after purification by silica gel chromatography (7:1 hexane:EtOAc) as a light yellow solid.

¹H NMR (CDCl₃, 500 MHz) δ 7.67-7.63 (m, 1H), 7.59-7.51 (m, 2H), 7.40-7.35 (m, 1H), 7.14-7.10 (m, 2H), 6.97-6.92 (m, 2H), 6.61 (dd, J = 8.7 Hz, 1.9 Hz, 1H), 6.29 (dd, J = 8.7 Hz, 1.9 Hz, 1H), 6.10-6.07 (m, 1H), 5.87 (s, 2H), 4.18-4.06 (m, 1H), 3.51-3.49 (m, 1H), 3.40-3.36 (m, 1H), 3.13-3.05 (m, 1H), 2.84-2.78 (m, 1H), 2.42-2.35 (m, 1H), 2.25-2.02 (m, 3H), 1.97-1.89 (m, 1H), 1.84-1.64 (m, 3H), 1.36-1.22 (m, 2H), 0.96-0.92 (m, 3H);

¹³C NMR (CDCl₃, 126 MHz) δ 162.4 (2), 160.5 (2), 154.4 (2), 151.6 (2), 148.1 (2), 141.5 (2), 139.8 (2), 136.6, 136.0, 131.4, 131.3, 128.8, 128.7, 128.2 (d, *J* = 170.2 Hz) (2), 123.8, 123.7, 115.4, 115.2, 107.8 (2), 105.7 (2), 101.1 (2), 98.2, 98.1, 69.7, 69.6, 62.7, 62.4, 54.6, 52.6, 52.1, 49.3, 44.3, 44.2, 42.4, 42.2, 34.5, 34.3, 32.6, 32.4, 19.8, 19.7, 14.3, 14.2;

¹⁹F NMR (CDCl₃, 470 MHz) δ -116.7;

HRMS (ESI): calcd for C₂₉H₃₁N₂O₅F [M+H]⁺: 507.2290. Found 507.2288.

5w Prepared according to the General Procedure A from Debenzyldonepezil (0.3 mmol, 1.5 equiv.), potassium aryl acetate (0.2 mmol, 1.0 equiv.), CuI (0.06 mmol, 0.3 equiv.), Zn(OAc)₂ (0.05 mmol, 0.25 equiv.), and MnO₂ (1.0 mmol, 5.0 equiv) in 1.0 mL DMF, 30 °C, 21h. 79% yield determined by ¹H NMR using Bn₂O as internal standard.

Prepared according to the General Procedure A from Debenzyldonepezil (1.5 mmol, 3.0 equiv.), potassium aryl acetate (0.5 mmol, 1.0 equiv.), CuI (0.15 mmol, 0.3 equiv.), Zn(OAc)₂ (0.125 mmol, 0.25 equiv.), and MnO₂ (2.5 mmol, 5.0 equiv) in 2.5 mL DMF, 30 °C, 21h. Isolated in 76% yield after purification by silica gel chromatography (1:2 hexane:EtOAc) as a white solid.

¹H NMR (CDCl₃, 400 MHz) δ 7.62 (d, *J* = 8.2 Hz, 1H), 7.57-7.49 (m, 2H), 7.34 (t, *J* = 8.2 Hz, 1H), 7.15 (s, 1H), 6.84 (s, 1H), 3.98-3.95 (m, 4H), 3.89 (s, 3H), 3.23-3.16 (m, 1H), 2.86-2.84 (m, 1H), 2.71-2.64 (m, 3H), 1.98-1.83 (m, 4H), 1.74 -1.56 (m, 3H), 1.39-1.16 (m, 6H), 0.89 (t, *J* = 7.7 Hz, 3H);

¹³C NMR (CDCl₃, 100 MHz) δ 207.9, 155.5, 151.6, 149.4, 148.7, 136.4, 131.4, 129.3, 129.0, 127.3, 123.5, 107.4, 104.4, 62.6, 56.2, 51.1, 49.8, 45.5, 38.6, 34.7, 33.2, 32.2, 32.0, 19.5, 14.3;

HRMS (ESI): calcd for C₂₇H₃₅N₂O₅ [M+H]⁺: 467.2540. Found 467.2548.

5x Prepared according to the General Procedure A from Desloratadine (0.3 mmol, 1.5 equiv.), potassium aryl acetate (0.2 mmol, 1.0 equiv.), CuI (0.06 mmol, 0.3 equiv.), Zn(OAc)₂

(0.05 mmol, 0.25 equiv.), and MnO₂ (1.0 mmol, 5.0 equiv) in 1.0 mL DMF, 30 °C, 44h. 87% yield determined by ¹H NMR using durene as internal standard.

Prepared according to the General Procedure A from Desloratadine (1.5 mmol, 3.0 equiv.), potassium aryl acetate (0.5 mmol, 1.0 equiv.), CuI (0.15 mmol, 0.3 equiv.), Zn(OAc)₂ (0.125 mmol, 0.25 equiv.), and MnO₂ (2.5 mmol, 5.0 equiv) in 2.5 mL DMF, 30 °C, 20h. Isolated in 92% yield after purification by silica gel chromatography (1:3 hexane:EtOAc) as a light yellow oil.

¹H NMR (CDCl₃, 700 MHz) δ 8.37-8.34 (m, 1H), 7.61-7.55 (m, 2H), 7.49 (t, *J* = 7.0 Hz, 1H), 7.39 (td, *J* = 7.4 Hz, 1.4 Hz, 1H), 7.33 (t, *J* = 7.0 Hz, 1H), 7.12-7.03 (m, 4H), 3.99-3.94 (m, 1H), 3.38-3.30 (m, 2H), 2.81-2.74 (m, 3H), 2.59-2.43 (m, 1H), 2.42-2.24 (m, 4H), 2.13-2.07 (m, 2H), 1.87-1.82 (m, 1H), 1.72-1.67 (m, 1H), 1.25-1.16 (m, 2H), 0.88-0.85 (m, 3H);

¹³C NMR (CDCl₃, 176 MHz) δ 157.7, 157.6, 151.4, 151.3, 146.6, 146.5, 139.5, 139.4, 139.2 (2), 137.8, 137.7, 137.2, 137.1, 136.5, 136.3, 133.4, 133.3, 132.6, 132.5, 132.4 (2), 131.5, 131.4, 130.9, 130.8, 128.9 (2), 128.8, 128.7, 127.4 (2), 125.9, 125.8, 123.5, 123.4, 122.0, 121.9, 62.3, 62.2, 52.0, 51.9, 51.1, 51.0, 33.5, 33.4, 31.8 (2), 31.4 (2), 31.2 (2), 31.0 (2), 19.4 (2), 14.2 (2);

HRMS (ESI): calcd for C₂₉H₃₁ClN₃O₂ [M+H]⁺: 488.2099. Found 488.2094.

5y Prepared according to the General Procedure A from Crizotinib (0.3 mmol, 1.5 equiv.), potassium aryl acetate (0.2 mmol, 1.0 equiv.), CuI (0.06 mmol, 0.3 equiv.), Zn(OAc)₂ (0.05 mmol, 0.25 equiv.), and MnO₂ (1.0 mmol, 5.0 equiv) in 1.0 mL DMF, 30 °C, 20h. 72% yield (1:1 dr) determined by ¹H NMR using Bn₂O as internal standard.

Prepared according to the General Procedure A from Crizotinib (1.5 mmol, 3.0 equiv.), potassium aryl acetate (0.5 mmol, 1.0 equiv.), CuI (0.15 mmol, 0.3 equiv.), Zn(OAc)₂ (0.125 mmol, 0.25 equiv.), and MnO₂ (2.5 mmol, 5.0 equiv) in 2.5 mL DMF, 30 °C, 20h. Isolated in 72% yield (1:1 dr) after purification by silica gel chromatography (10:1 DCM:MeOH) as a light yellow solid.

¹H NMR (CDCl₃, 500 MHz) δ 7.75 (d, *J* = 1.6 Hz, 1H), 7.66 (dd, *J* = 7.7 Hz, 1.1 Hz, 1H), 7.58-7.52 (m, 3H), 7.46 (s, 1H), 7.38 (td, *J* = 8.1 Hz, 1.6 Hz, 1H), 7.32-7.29 (m, 1H), 7.05 (t, *J* = 7.2 Hz,

1H), 6.86 (d, J = 1.6 Hz, 1H), 6.06 (q, J = 6.7 Hz, 1H), 4.74 (s, 2H), 4.08-3.99 (m, 2H), 2.96 (d, J = 9.6 Hz, 1H), 2.86 (d, J = 9.6 Hz, 1H), 2.27-2.15 (m, 2H), 2.09-2.02 (m, 2H), 1.96-1.87 (m, 2H), 1.85 (d, J = 6.1 Hz, 3H), 1.78-1.71 (m, 1H), 1.64 (brs, 1H), 1.32-1.18 (m, 2H), 0.92 (t, J = 7.6 Hz, 3H);

^{13}C NMR (CDCl_3 , 176 MHz) δ 151.4, 148.8, 139.8, 137.0, 136.3 (d, J = 248.2 Hz), 136.2, 135.5, 131.5, 129.8, 128.9 (d, J = 3.9 Hz), 128.7, 127.6, 123.7, 122.5, 122.2 (d, J = 19.5 Hz), 119.8, 119.3, 116.7 (d, J = 24.0 Hz), 116.6, 115.6, 72.4, 62.2, 59.6, 50.0, 47.9, 33.0, 32.9, 32.6, 19.5, 18.9, 14.3;

^{19}F NMR (CDCl_3 , 470 MHz) δ -112.1;

HRMS (ESI): calcd for $\text{C}_{31}\text{H}_{34}\text{N}_6\text{O}_3\text{FCl}_2$ $[\text{M}+\text{H}]^+$: 627.2048. Found 627.2052.

5z Prepared according to the General Procedure A from Fasudil (0.3 mmol, 1.5 equiv.), potassium aryl acetate (0.2 mmol, 1.0 equiv.), CuI (0.06 mmol, 0.3 equiv.), $\text{Zn}(\text{OAc})_2$ (0.05 mmol, 0.25 equiv.), and MnO_2 (1.0 mmol, 5.0 equiv) in 1.0 mL DMF, 30 °C, 44h. 57% yield determined by ^1H NMR using durene as internal standard.

Prepared according to the General Procedure A from Fasudil (1.5 mmol, 3.0 equiv.), potassium aryl acetate (0.5 mmol, 1.0 equiv.), CuI (0.15 mmol, 0.3 equiv.), $\text{Zn}(\text{OAc})_2$ (0.125 mmol, 0.25 equiv.), and MnO_2 (2.5 mmol, 5.0 equiv) in 2.5 mL DMF, 30 °C, 45h. Isolated in 86% yield after purification by silica gel chromatography (1:2 hexane:EtOAc) as a yellow oil.

^1H NMR (CDCl_3 , 700 MHz) δ 9.32 (s, 1H), 8.65 (d, J = 6.2 Hz, 1H), 8.40 (d, J = 6.2 Hz, 1H), 8.24 (dd, J = 7.4 Hz, 1.0 Hz, 1H), 8.16 (d, J = 8.5 Hz, 1H), 7.66 (t, J = 7.8 Hz, 1H), 7.55 (dd, J = 7.8 Hz, 1.0 Hz, 1H), 7.47 (td, J = 7.8 Hz, 1.0 Hz, 1H), 7.36-7.33 (m, 2H), 4.32 (dd, J = 8.7 Hz, 5.9 Hz, 1H), 3.38-3.31 (m, 3H), 3.22-3.20 (m, 1H), 2.67-2.63 (m, 4H), 1.79-1.73 (m, 3H), 1.61-1.59 (m, 1H), 1.32-1.25 (m, 2H), 0.96 (t, J = 7.4 Hz, 3H);

^{13}C NMR (CDCl_3 , 176 MHz) δ 153.2, 151.5, 145.1, 136.1, 134.8, 133.2, 132.5, 131.7, 131.3, 129.2, 127.8(2), 125.8, 124.1, 117.7, 62.5, 53.4, 50.6, 49.7, 46.9, 29.7, 28.6, 20.3, 14.3;

HRMS (ESI): calcd for $\text{C}_{24}\text{H}_{29}\text{N}_4\text{O}_4\text{S}$ $[\text{M}+\text{H}]^+$: 469.1904. Found 469.1903.

5aa Prepared according to the General Procedure B from (S)-Duloxetine (0.75 mmol, 1.5 equiv.), aryl acetic acid (0.5 mmol, 1.0 equiv.), K_2CO_3 (0.5 mmol, 1.0 equiv.), CuI (0.15 mmol, 0.3 equiv.), and MnO_2 (2.5 mmol, 5.0 equiv) in 2.5 mL DMF, rt, 42h. Isolated in 84% yield (1:1 dr) after purification by silica gel chromatography (1:4 hexane:EtOAc) as a yellow oil.

1H NMR ($CDCl_3$, 500 MHz) δ 8.22-8.15 (m, 1H), 7.81-7.79 (m, 1H), 7.50-7.19 (m, 7H), 7.13-6.91 (m, 4H), 6.86-6.79 (m, 1H), 5.74-5.57 (m, 1H), 4.21-4.01 (m, 1H), 2.76-2.54 (m, 1H), 2.53-2.31 (m, 1H), 2.28-2.27 (m, 4H), 2.14-2.10 (m, 1H), 1.85-1.63 (m, 2H), 1.21-1.14 (m, 2H), 0.87-0.78 (m, 3H);

^{13}C NMR ($CDCl_3$, 126 MHz) δ 153.4, 153.3, 151.3, 151.2, 146.0, 145.7, 136.4, 136.1, 134.6, 134.5, 131.4, 131.0, 128.7, 128.1, 127.4 (2), 127.1, 127.0, 126.6, 126.5, 126.3, 126.2, 126.1, 126.0, 125.8 (2), 125.5, 124.9, 124.5, 124.4, 124.3 (2), 123.7, 123.2, 122.2, 122.1, 120.3, 120.2, 106.9, 106.7, 73.6, 73.5, 62.4, 61.7, 49.9, 48.5, 39.3, 38.9, 37.3, 37.2, 33.8, 30.6, 19.8, 19.4, 14.2 (2);

HRMS (ESI): calcd for $C_{28}H_{31}N_2O_3S$ $[M+H]^+$: 475.2050. Found 475.2051.

6a Prepared according to the General Procedure A from piperidine (1.0 mmol, 2.0 equiv.), the corresponding potassium aryl acetate (0.5 mmol, 1.0 equiv.), $Cu(OAc)_2$ (0.15 mmol, 0.3 equiv.), and MnO_2 (2.5 mmol, 5.0 equiv) in 2.5 mL DMF, rt, 20h. Isolated in 47% yield after purification by silica gel chromatography (1:1 hexane:EtOAc) as a yellow oil. Spectroscopic data agreed with that reported.⁴

1H NMR ($CDCl_3$, 700 MHz) δ 8.16 (d, J = 8.9 Hz, 2H), 7.50 (d, J = 8.9 Hz, 2H), 3.54 (s, 2H), 2.38 (brs, 4H), 1.59-1.56 (m, 4H), 1.45 (brs, 2H);

^{13}C NMR ($CDCl_3$, 176 MHz) δ 147.1, 147.0, 129.4, 123.4, 62.9, 54.6, 25.9, 24.2.

6b Prepared according to the General Procedure A from piperidine (1.0 mmol, 2.0 equiv.), the corresponding potassium aryl acetate (0.5 mmol, 1.0 equiv.), CuBr₂ (0.05 mmol, 0.1 equiv.), and MnO₂ (2.5 mmol, 5.0 equiv) in 2.5 mL DMA, 60 °C, 16h. Isolated in 58% yield after purification by silica gel chromatography (1:2 hexane:EtOAc) as a light yellow solid. Spectroscopic data agreed with that reported.⁵

¹H NMR (CDCl₃, 700 MHz) δ 7.59 (dd, *J* = 8.2 Hz, 1.8 Hz, 2H), 7.44 (d, *J* = 8.0 Hz, 2H), 3.50 (s, 2H), 2.36 (brs, 4H), 1.59-1.56 (m, 4H), 1.44 (brs, 2H);

¹³C NMR (CDCl₃, 176 MHz) δ 144.9, 132.0, 129.4, 119.1, 110.6, 63.3, 54.6, 25.9, 24.2.

6c Prepared according to the General Procedure A from piperidine (1.0 mmol, 2.0 equiv.), the corresponding potassium aryl acetate (0.5 mmol, 1.0 equiv.), CuBr₂ (0.05 mmol, 0.1 equiv.), and MnO₂ (2.5 mmol, 5.0 equiv) in 2.5 mL DMA, 60 °C, 16h. Isolated in 64% yield after purification by silica gel chromatography (10:1 DCM:MeOH) as a white solid. Spectroscopic data agreed with that reported.⁵

¹H NMR (CDCl₃, 500 MHz) δ 7.87 (d, *J* = 8.3 Hz, 2H), 7.54 (d, *J* = 8.3 Hz, 2H), 3.54 (s, 2H), 3.05 (s, 3H), 2.37 (brs, 4H), 1.59-1.57 (m, 4H), 1.47-1.43 (m, 2H);

¹³C NMR (CDCl₃, 126 MHz) δ 145.9, 139.0, 129.7, 127.3, 63.1, 54.6, 44.6, 26.0, 24.2.

6d Prepared according to the General Procedure A from piperidine (2.5 mmol, 5.0 equiv.), the corresponding potassium aryl acetate (0.5 mmol, 1.0 equiv.), CuBr₂ (0.025 mmol, 0.05 equiv.), and MnO₂ (2.5 mmol, 5.0 equiv) in 2.5 mL DMA, 60 °C, 22h. Isolated in 43% yield after purification by silica gel chromatography (20:1 DCM:MeOH) as a yellow oil. Spectroscopic data agreed with that reported.⁵

¹H NMR (CDCl₃, 700 MHz) δ 7.90 (dd, *J* = 7.0 Hz, 2.1 Hz, 2H), 7.42 (d, *J* = 8.4 Hz, 2H), 3.51 (s, 2H), 2.59 (s, 3H), 2.37 (brs, 4H), 1.58-1.55 (m, 4H), 1.42 (brs, 2H);

¹³C NMR (CDCl₃, 176 MHz) δ 197.9, 144.7, 135.9, 129.1, 128.3, 63.9, 54.6, 26.6, 25.9, 24.3.

6e Prepared according to the General Procedure B from piperidine (2.5 mmol, 5.0 equiv.), the corresponding aryl acetic acid (0.5 mmol, 1.0 equiv.), CuBr₂ (0.025 mmol, 0.05 equiv.), K₂CO₃ (0.5 mmol, 1.0 equiv.), and MnO₂ (2.5 mmol, 5.0 equiv) in 2.5 mL DMA, 60 °C, 15h. Isolated in 45% yield after purification by silica gel chromatography (20:1 DCM:MeOH) as a yellow oil.

¹H NMR (CDCl₃, 700 MHz) δ 7.80-7.78 (m, 2H), 7.75 (d, *J* = 8.2 Hz, 2H), 7.58-7.56 (m, 1H), 7.47 (t, *J* = 7.7 Hz, 2H), 7.43 (d, *J* = 8.3 Hz, 2H), 3.53 (s, 2H), 2.39 (brs, 4H), 1.60-1.57 (m, 4H), 1.44 (brs, 2H);

¹³C NMR (CDCl₃, 176 MHz) δ 196.5, 144.0, 137.9, 136.2, 132.3, 130.1, 130.0, 128.9, 128.2, 63.5, 54.6, 26.0, 24.3;

HRMS (ESI): calcd for C₁₉H₂₂NO [M+H]⁺: 280.1696. Found 280.1696.

6f Prepared according to the General Procedure A from piperidine (1.0 mmol, 2.0 equiv.), the corresponding potassium aryl acetate (0.5 mmol, 1.0 equiv.), Cu(OAc)₂ (0.15 mmol, 0.3 equiv.), and MnO₂ (2.5 mmol, 5.0 equiv) in 2.5 mL DMF, rt, 44h. Isolated in 62% yield after purification by silica gel chromatography (5:1 hexane:EtOAc) as a light yellow oil. Spectroscopic data agreed with that reported.⁶

¹H NMR (CDCl₃, 700 MHz) δ 7.80 (d, *J* = 8.4 Hz, 1H), 7.64 (d, *J* = 7.5 Hz, 1H), 7.52 (t, *J* = 7.5 Hz, 1H), 7.36 (t, *J* = 7.5 Hz, 1H), 3.73 (s, 2H), 2.36 (brs, 4H), 1.56-1.52 (m, 4H), 1.42 (brs, 2H);

¹³C NMR (CDCl₃, 176 MHz) δ 149.8, 134.7, 132.2, 130.8, 127.5, 124.2, 59.6, 54.6, 26.0, 24.2.

6g Prepared according to the General Procedure B from piperidine (2.5 mmol, 5.0 equiv.), the corresponding aryl acetic acid (0.5 mmol, 1.0 equiv.), CuBr₂ (0.05 mmol, 0.1 equiv.), K₂CO₃ (0.5 mmol, 1.0 equiv.), and MnO₂ (2.5 mmol, 5.0 equiv) in 2.5 mL DMA, 70 °C, 23h. Isolated in

46% yield after purification by silica gel chromatography (3:1 hexane:EtOAc) as a light yellow oil.

¹H NMR (CDCl₃, 700 MHz) δ 7.62 (dd, *J* = 7.6 Hz, 1.3 Hz, 1H), 7.57 (dd, *J* = 7.6 Hz, 1.3 Hz, 1H), 7.53 (td, *J* = 7.6 Hz, 1.3 Hz, 1H), 7.32 (td, *J* = 7.6 Hz, 1.4 Hz, 1H), 3.66 (s, 2H), 2.43 (brs, 4H), 1.59-1.55 (m, 4H), 1.45-1.42 (m, 2H);

¹³C NMR (CDCl₃, 176 MHz) δ 143.2, 132.7, 132.4, 129.9, 127.3, 117.9, 112.8, 61.2, 54.5, 25.9, 24.2;

HRMS (ESI): calcd for C₁₃H₁₇N₂ [M+H]⁺: 201.1386. Found 201.1386.

6h Prepared according to the General Procedure A from piperidine (1.0 mmol, 2.0 equiv.), the corresponding potassium aryl acetate (0.5 mmol, 1.0 equiv.), CuI (0.15 mmol, 0.3 equiv.), and MnO₂ (2.5 mmol, 5.0 equiv) in 2.5 mL DMA, 60 °C. Isolated in 53% yield after purification by silica gel chromatography (10:1 DCM:MeOH) as a yellow oil. Spectroscopic data agreed with that reported.⁷

¹H NMR (CDCl₃, 500 MHz) δ 8.52 (d, *J* = 5.2 Hz, 2H), 7.26 (t, *J* = 2.2 Hz, 2H), 3.45 (s, 2H), 2.37 (brs, 4H), 1.61-1.56 (m, 4H), 1.46-1.44 (m, 2H);

¹³C NMR (CDCl₃, 126 MHz) δ 149.7, 148.1, 123.9, 62.6, 54.7, 26.0, 24.2.

6i Prepared according to the General Procedure A from piperidine (1.0 mmol, 2.0 equiv.), the corresponding potassium aryl acetate (0.5 mmol, 1.0 equiv.), Cu(OAc)₂ (0.15 mmol, 0.3 equiv.), and MnO₂ (2.5 mmol, 5.0 equiv) in 2.5 mL DMF, rt, 18h. Isolated in 61% yield after purification by silica gel chromatography (1:2 hexane:EtOAc) as a yellow oil.

¹H NMR (CDCl₃, 500 MHz) δ 7.83 (dd, *J* = 8.2 Hz, 2.3 Hz, 1H), 7.68 (d, *J* = 2.2 Hz, 1H), 7.34 (brs, 1H), 3.91 (s, 3H), 3.57 (s, 2H), 2.45 (brs, 4H), 1.62 (m, 4H), 1.46 (brs, 2H);

¹³C NMR (CDCl₃, 126 MHz) δ 157.7, 147.4, 135.1, 129.8, 115.7, 105.0, 56.4, 55.8, 54.8, 26.1, 24.2;

HRMS (ESI): calcd for C₁₃H₁₉N₂O₃ [M+H]⁺: 251.1390. Found 251.1393.

6j Prepared according to the General Procedure A from piperidine (1.5 mmol, 3.0 equiv.), the corresponding potassium aryl acetate (0.5 mmol, 1.0 equiv.), CuI (0.15 mmol, 0.3 equiv.), Zn(OAc)₂ (0.5 mmol, 1.0 equiv.), and MnO₂ (2.5 mmol, 5.0 equiv) in 2.5 mL DMF, rt, 17h. Isolated in 72% yield after purification by silica gel chromatography (12:1 hexane:EtOAc) as a light yellow oil.

¹H NMR (CDCl₃, 500 MHz) δ 7.76 (d, *J* = 2.1 Hz, 1H), 7.62 (dd, *J* = 8.5 Hz, 2.1 Hz, 1H), 7.43 (d, *J* = 8.5 Hz, 1H), 3.89 (q, *J* = 4.9 Hz, 1H), 2.36-2.32 (m, 2H), 2.28-2.24 (m, 2H), 1.88-1.81 (m, 1H), 1.70-1.63 (m, 1H), 1.48-1.37 (m, 4H), 1.36-1.27 (m, 2H), 1.25-1.13 (m, 2H), 0.89 (t, *J* = 7.4 Hz, 3H);

¹³C NMR (CDCl₃, 126 MHz) δ 151.8, 135.9, 134.4, 130.5, 126.5, 120.1, 62.8, 51.0, 32.7, 26.2, 24.6, 19.5, 14.2;

HRMS (ESI): calcd for C₁₅H₂₂BrN₂O₂ [M+H]⁺: 341.0859. Found 341.0858.

6k Prepared according to the General Procedure A from piperidine (1.5 mmol, 3.0 equiv.), the corresponding potassium aryl acetate (0.5 mmol, 1.0 equiv.), CuI (0.15 mmol, 0.3 equiv.), and MnO₂ (2.5 mmol, 5.0 equiv) in 2.5 mL DMF, 40 °C, 24h. Isolated in 91% yield after purification by silica gel chromatography (2:1 hexane:EtOAc) as a light yellow oil.

¹H NMR (CDCl₃, 700 MHz) δ 7.43 (d, *J* = 8.7 Hz, 1H), 7.13 (d, *J* = 3.0 Hz, 1H), 7.05 (dd, *J* = 8.8 Hz, 2.8 Hz, 1H), 3.87 (q, *J* = 4.4 Hz, 1H), 3.84 (s, 3H), 2.33-2.26 (m, 4H), 1.86-1.82 (m, 1H), 1.71-1.65 (m, 1H), 1.47-1.44 (m, 4H), 1.35-1.33 (m, 2H), 1.26-1.16 (m, 2H), 0.88 (t, *J* = 7.7 Hz, 3H);

¹³C NMR (CDCl₃, 176 MHz) δ 158.2, 152.0, 129.9, 128.1, 117.9, 108.2, 62.4, 55.7, 51.0, 33.2, 26.2, 24.7, 19.5, 14.3;

HRMS (ESI): calcd for C₁₆H₂₅N₂O₃ [M+H]⁺: 293.1860. Found 293.1859.

6l Prepared according to the General Procedure A from piperidine (1.5 mmol, 3.0 equiv.), the corresponding potassium aryl acetate (0.5 mmol, 1.0 equiv.), CuI (0.15 mmol, 0.3 equiv.), and MnO₂ (2.5 mmol, 5.0 equiv) in 2.5 mL DMF, 60 °C, 24h. Isolated in 87% yield after purification by silica gel chromatography (1:3 hexane:EtOAc) as a yellow oil.

¹H NMR (CDCl₃, 500 MHz) δ 7.32 (d, *J* = 8.8 Hz, 1H), 6.87 (d, *J* = 2.8 Hz, 1H), 6.82 (dd, *J* = 8.7 Hz, 2.7 Hz, 1H), 3.81 (q, *J* = 4.3 Hz, 1H), 2.98 (s, 6H), 2.34-2.26 (m, 4H), 1.87-1.80 (m, 1H), 1.73-1.65 (m, 1H), 1.49-1.44 (m, 4H), 1.36-1.15 (m, 4H), 0.88 (t, *J* = 7.5 Hz, 3H);

¹³C NMR (CDCl₃, 126 MHz) δ 152.6, 149.1, 129.5, 122.4, 114.9, 106.2, 62.3, 51.0, 40.2, 33.5, 26.3, 24.7, 19.6, 14.3;

HRMS (ESI): calcd for C₁₇H₂₈N₃O₂ [M+H]⁺: 306.2176. Found 306.2173.

6m Prepared according to the General Procedure A from piperidine (1.5 mmol, 3.0 equiv.), the corresponding potassium aryl acetate (0.5 mmol, 1.0 equiv.), CuI (0.15 mmol, 0.3 equiv.), Zn(OAc)₂ (0.5 mmol, 1.0 equiv.), and MnO₂ (2.5 mmol, 5.0 equiv) in 2.5 mL DMF, rt, 18h. Isolated in 67% yield after purification by silica gel chromatography (15:1 hexane:EtOAc) as a light yellow oil.

¹H NMR (CDCl₃, 500 MHz) δ 7.61 (d, *J* = 8.8 Hz, 1H), 7.53 (d, *J* = 2.3 Hz, 1H), 7.31 (dd, *J* = 8.8 Hz, 2.3 Hz, 1H), 3.97 (q, *J* = 4.7 Hz, 1H), 2.38-2.25 (m, 4H), 1.88-1.81 (m, 1H), 1.69-1.62 (m, 1H), 1.49-1.43 (m, 4H), 1.38-1.33 (m, 2H), 1.29-1.17 (m, 2H), 0.90 (t, *J* = 7.4 Hz, 3H);

¹³C NMR (CDCl₃, 126 MHz) δ 149.7, 139.3, 137.8, 129.0, 127.4, 125.1, 62.9, 51.1, 32.9, 26.2, 24.6, 19.4, 14.2;

HRMS (ESI): calcd for C₁₅H₂₂ClN₂O₂ [M+H]⁺: 297.1364. Found 297.1367.

6n Prepared according to the General Procedure B from morpholine (12.0 mmol, 3.0 equiv.), the corresponding aryl acetic acid (4.0 mmol, 1.0 equiv.), CuI (1.2 mmol, 0.3 equiv.), Zn(OAc)₂ (4.0 mmol, 1.0 equiv.), K₂CO₃ (4.0 mmol, 1.0 equiv.), and MnO₂ (20.0 mmol, 5.0 equiv) in 20 mL DMF, rt, 53h. Isolated in 82% yield after purification by silica gel chromatography (10:1 hexane:EtOAc) as a light yellow oil. For 0.5 mmol scale, the product was afforded in 86% yield.

¹H NMR (CDCl₃, 400 MHz) δ 7.64 (d, *J* = 8.8 Hz, 1H), 7.60 (d, *J* = 2.2 Hz, 1H), 7.34 (dd, *J* = 8.7 Hz, *J* = 2.2 Hz, 1H), 3.93 (q, *J* = 4.7 Hz, 1H), 3.66-3.57 (m, 4H), 2.51-2.46 (m, 2H), 2.37-2.31 (m, 2H), 1.90-1.83 (m, 1H), 1.69-1.59 (m, 1H), 1.25-1.13 (m, 2H), 0.89 (t, *J* = 7.3 Hz, 3H);

¹³C NMR (CDCl₃, 126 MHz) δ 149.6, 138.7, 138.2, 129.2, 127.8, 125.2, 67.1, 63.0, 50.8, 33.3, 19.1, 14.2;

HRMS (ESI): calcd for C₁₄H₂₀ClN₂O₃ [M+H]⁺: 299.1157. Found 299.1157.

6o Prepared according to the General Procedure A from piperidine (1.5 mmol, 3.0 equiv.), the corresponding potassium aryl acetate (0.5 mmol, 1.0 equiv.), CuI (0.15 mmol, 0.3 equiv.), Zn(OAc)₂ (0.125 mmol, 0.25 equiv.), and MnO₂ (2.5 mmol, 5.0 equiv) in 2.5 mL DMF, 30 °C, 26h. Isolated in 79% yield after purification by silica gel chromatography (4:1 hexane:EtOAc) as a light yellow oil.

¹H NMR (CDCl₃, 700 MHz) δ 7.77 (d, *J* = 9.0 Hz, 1H), 7.10 (d, *J* = 2.5 Hz, 1H), 6.79 (dd, *J* = 8.8 Hz, 2.5 Hz, 1H), 4.15 (q, *J* = 4.5 Hz, 1H), 3.88 (s, 3H), 2.37-2.28 (m, 4H), 1.86-1.82 (m, 1H), 1.69-1.66 (m, 1H), 1.48-1.46 (m, 4H), 1.36-1.35 (m, 2H), 1.25-1.19 (m, 2H), 0.88 (t, *J* = 7.4 Hz, 3H);

¹³C NMR (CDCl₃, 176 MHz) δ 162.0, 144.5, 140.3, 126.4, 114.2, 111.7, 62.7, 55.7, 51.2, 33.7, 26.3, 24.7, 19.2, 14.3;

HRMS (ESI): calcd for C₁₆H₂₅N₂O₃ [M+H]⁺: 293.1860. Found 293.1863.

6p Prepared according to the General Procedure A from the corresponding amine (1.5 mmol, 3.0 equiv.), potassium aryl acetate (0.5 mmol, 1.0 equiv.), CuI (0.15 mmol, 0.3 equiv.), and MnO₂ (2.5 mmol, 5.0 equiv) in 2.5 mL DMF, 30 °C, 18h. Isolated in 73% yield after purification by silica gel chromatography (10:1 hexane:EtOAc) as a light yellow oil.

¹H NMR (CDCl₃, 500 MHz) δ 7.61 (t, *J* = 7.4 Hz, 2H), 7.49 (td, *J* = 7.4 Hz, 1.3 Hz, 1H), 7.33 (td, *J* = 7.4 Hz, 1.4 Hz, 1H), 3.99 (q, *J* = 6.8 Hz, 1H), 2.38-2.34 (m, 2H), 2.28-2.23 (m, 2H), 1.52-1.40 (m, 4H), 1.39-1.34 (m, 5H);

¹³C NMR (CDCl₃, 126 MHz) δ 150.5, 139.5, 131.7, 128.4, 127.3, 123.6, 58.7, 50.9, 26.0, 24.6, 16.0;

HRMS (ESI): calcd for C₁₃H₁₉N₂O₂ [M+H]⁺: 235.1441. Found 235.1439.

6q Prepared according to the General Procedure A from piperidine (1.0 mmol, 2.0 equiv.), the corresponding potassium aryl acetate (0.5 mmol, 1.0 equiv.), CuI (0.15 mmol, 0.3 equiv.), Zn(OAc)₂ (0.5 mmol, 1.0 equiv.), and MnO₂ (2.5 mmol, 5.0 equiv) in 2.5 mL DMF/DCE (1:1), rt, 20h. Isolated in 75% yield after purification by silica gel chromatography (10:1 hexane:EtOAc) as a light yellow oil.

¹H NMR (CDCl₃, 700 MHz) δ 7.89 (s, 1H), 7.80-7.75 (m, 2H), 4.05-4.02 (m, 1H), 2.37 (brs, 2H), 2.26 (brs, 2H), 1.49-1.45 (m, 4H), 1.38-1.35 (m, 5H);

¹³C NMR (CDCl₃, 176 MHz) δ 150.2, 143.9, 130.0 (q, *J* = 34.2 Hz), 129.4, 128.2, 123.0 (q, *J* = 272.6 Hz), 121.0, 58.7, 50.9, 25.4, 24.5, 15.8;

¹⁹F NMR (CDCl₃, 470 MHz) δ -62.8;

HRMS (ESI): calcd for C₁₄H₁₈F₃N₂O₂ [M+H]⁺: 303.1315. Found 303.1314.

6r Prepared according to the General Procedure A from piperidine (1.5 mmol, 3.0 equiv.), the corresponding potassium aryl acetate (0.5 mmol, 1.0 equiv.), CuI (0.15 mmol, 0.3 equiv.), and MnO₂ (2.5 mmol, 5.0 equiv) in 2.5 mL DMF, 30 °C, 21h. Isolated in 86% yield after purification by silica gel chromatography (1:3 hexane:EtOAc) as a light yellow oil.

¹H NMR (CDCl₃, 500 MHz) δ 7.71 (d, *J* = 8.0 Hz, 1H), 7.56 (t, *J* = 8.0 Hz, 1H), 7.47-7.39 (m, 2H), 4.87 (brs, 1H), 4.53 (dd, *J* = 9.5 Hz, 4.0 Hz, 1H), 3.92-3.81 (m, 2H), 2.49-2.41 (m, 3H), 2.22 (brs, 2H), 1.77-1.72 (m, 1H), 1.57-1.49 (m, 4H), 1.32-1.28 (m, 2H);

¹³C NMR (CDCl₃, 126 MHz) δ 151.1, 131.5, 131.4, 129.6, 128.1, 123.9, 62.9, 62.6, 50.6, 31.8, 26.3, 24.2;

HRMS (ESI): calcd for C₁₄H₂₁N₂O₃ [M+H]⁺: 265.1547. Found 265.1545.

6s Prepared according to the General Procedure A from piperidine (1.5 mmol, 3.0 equiv.), the corresponding potassium aryl acetate (0.5 mmol, 1.0 equiv.), CuI (0.15 mmol, 0.3 equiv.), Zn(OAc)₂ (0.125 mmol, 0.25 equiv.), and MnO₂ (2.5 mmol, 5.0 equiv) in 2.5 mL DMF, 30 °C, 22h. Isolated in 81% yield after purification by silica gel chromatography (7:1 hexane:EtOAc) as a light yellow oil.

¹H NMR (CDCl₃, 700 MHz) δ 7.63 (d, *J* = 8.0 Hz, 1H), 7.49-7.47 (m, 2H), 7.36-7.33 (m, 1H), 4.18 (q, *J* = 5.4 Hz, 1H), 3.64-3.56 (m, 2H), 2.32-2.30 (m, 4H), 2.19-2.17 (m, 1H), 1.97-1.94 (m, 1H), 1.45 (brs, 4H), 1.35-1.32 (m, 2H), 0.87 (s, 9H), 0.01 (s, 6H);

¹³C NMR (CDCl₃, 176 MHz) δ 151.4, 135.8, 131.2, 128.8, 127.4, 123.8, 60.7, 59.6, 50.7, 33.2, 26.2, 25.9, 24.6, 18.3, -5.4;

HRMS (ESI): calcd for C₂₀H₃₅N₂O₃Si [M+H]⁺: 379.2411. Found 379.2418.

6u Prepared according to the General Procedure B from piperidine (1.0 mmol, 3.0 equiv.), the corresponding aryl acetic acid (0.5 mmol, 1.0 equiv.), CuI (0.15 mmol, 0.3 equiv.), Zn(OAc)₂ (0.5 mmol, 1.0 equiv.), and MnO₂ (2.5 mmol, 5.0 equiv) in 2.5 mL DMF, rt, 72h. Isolated in 71% yield after purification by silica gel chromatography (4:1 hexane:EtOAc) as a yellow solid.

¹H NMR (CDCl₃, 500 MHz) δ 7.64 (d, *J* = 2.1 Hz, 1H), 7.51 (d, *J* = 8.6 Hz, 1H), 7.47-7.45 (m, 1H), 4.55 (dd, *J* = 9.1 Hz, 3.1 Hz, 1H), 3.91-3.83 (m, 3H), 3.78-3.74 (m, 1H), 2.31-2.28 (m, 5H), 2.18-2.13 (m, 1H), 1.47-1.35 (m, 4H), 1.35-1.31 (m, 5H);

¹³C NMR (CDCl₃, 126 MHz) δ 150.1, 138.7, 132.3, 130.2, 125.4, 125.3, 109.0, 64.6, 64.5, 57.9, 50.2, 37.4, 26.1, 24.7, 24.5;

HRMS (ESI): calcd for C₁₇H₂₄BrN₂O₄ [M+H]⁺: 399.0914. Found 399.0912.

6v Prepared according to the General Procedure A from piperidine (1.0 mmol, 2.0 equiv.), the corresponding potassium aryl acetate (0.5 mmol, 1.0 equiv.), Cu(OAc)₂ (0.25 mmol, 0.5 equiv.), and MnO₂ (2.5 mmol, 5.0 equiv) in 2.5 mL DMF, rt, 18h. Isolated in 88% yield after purification by silica gel chromatography (1:1 hexane:EtOAc) as a yellow oil.

¹H NMR (CDCl₃, 400 MHz) δ 7.82 (dd, *J* = 8.8 Hz, 2.4 Hz, 1H), 7.68 (d, *J* = 2.4 Hz, 1H), 7.64 (d, *J* = 8.4 Hz, 1H), 3.90 (s, 3H), 3.88 (q, *J* = 6.8 Hz, 1H), 2.45-2.42 (m, 2H), 2.31-2.26 (m, 2H), 1.56-1.49 (m, 4H), 1.42-1.36 (m, 2H), 1.24 (d, *J* = 7.0 Hz, 3H);

¹³C NMR (CDCl₃, 100 MHz) δ 157.2, 147.3, 141.6, 128.1, 115.9, 105.3, 56.7, 55.9, 51.8, 26.3, 24.6, 19.6;

HRMS (ESI): calcd for C₁₄H₂₁N₂O₃ [M+H]⁺: 265.1547. Found 265.1547.

6w Prepared according to the General Procedure A from piperidine (1.0 mmol, 2.0 equiv.), the corresponding potassium aryl acetate (0.5 mmol, 1.0 equiv.), Cu(OAc)₂ (0.25 mmol, 0.5 equiv.), and MnO₂ (2.5 mmol, 5.0 equiv) in 2.5 mL DMF/DCE (1:1), rt, 18h. Isolated in 84% yield after purification by silica gel chromatography (2:1 hexane:EtOAc) as a light yellow oil.

¹H NMR (CDCl₃, 500 MHz) δ 7.85-7.81 (m, 2H), 7.69 (dd, *J* = 8.6 Hz, 1.8 Hz, 1H), 3.51 (q, *J* = 6.8 Hz, 1H), 2.44-2.42 (m, 2H), 2.32-2.27 (m, 2H), 1.58-1.52 (m, 4H), 1.45-1.40 (m, 2H), 1.34 (d, *J* = 6.8 Hz, 3H);

¹³C NMR (CDCl₃, 126 MHz) δ 151.5, 146.6, 131.7, 126.9 (q, *J* = 5.2 Hz), 125.1, 123.4 (q, *J* = 63.5 Hz), 122.2 (d, *J* = 273.9 Hz), 64.1, 51.4, 26.2, 24.4, 18.7;

¹⁹F NMR (CDCl₃, 470 MHz) δ -59.8;

HRMS (ESI): calcd for C₁₄H₁₈F₃N₂O₂ [M+H]⁺: 303.1315. Found 303.1314.

6x Prepared according to the General Procedure A from piperidine (1.0 mmol, 2.0 equiv.), the corresponding potassium aryl acetate (0.5 mmol, 1.0 equiv.), Cu(OAc)₂ (0.25 mmol, 0.5 equiv.), and MnO₂ (2.5 mmol, 5.0 equiv) in 2.5 mL DMF, rt, 22h. Isolated in 74% yield after purification by silica gel chromatography (1:1 hexane:EtOAc) as a light yellow oil.

Prepared according to the General Procedure B from piperidine (10.0 mmol, 2.0 equiv.), the corresponding aryl acetic acid (5 mmol, 1.0 equiv.), CuI (2.5 mmol, 0.5 equiv.), K₂CO₃ (0.5 mmol, 1.0 equiv.), and MnO₂ (2.5 mmol, 5.0 equiv) in 25 mL DMF, rt, 24h. Isolated in 79% yield after purification by silica gel chromatography.

¹H NMR (CDCl₃, 400 MHz) δ 8.16 (d, *J* = 8.6 Hz, 2H), 7.39 (d, *J* = 8.6 Hz, 2H), 3.39 (q, *J* = 4.8 Hz, 1H), 2.33-2.30 (m, 4H), 1.91-1.67 (m, 2H), 1.58-1.51 (m, 4H), 1.38-1.24 (m, 2H), 1.19-1.02 (m, 2H), 0.85 (t, *J* = 7.4 Hz, 3H);

¹³C NMR (CDCl₃, 100 MHz) δ 149.2, 146.9, 129.2, 123.1, 69.7, 51.4, 34.7, 26.3, 24.6, 19.4, 14.1;

HRMS (ESI): calcd for C₁₅H₂₃N₂O₂ [M+H]⁺: 263.1754. Found 263.1752.

6y Prepared according to the General Procedure A from piperidine (1.0 mmol, 2.0 equiv.), the corresponding potassium aryl acetate (0.5 mmol, 1.0 equiv.), Cu(OAc)₂ (0.25 mmol, 0.5 equiv.), and MnO₂ (2.5 mmol, 5.0 equiv) in 2.5 mL DMF, rt, 22h. Isolated in 67% yield after purification by silica gel chromatography (1:1 hexane:EtOAc) as a light yellow oil.

¹H NMR (CDCl₃, 400 MHz) δ 8.16 (d, *J* = 8.6 Hz, 2H), 7.50 (d, *J* = 8.6 Hz, 2H), 3.48 (q, *J* = 6.8 Hz, 1H), 2.42-2.26 (m, 4H), 1.58-1.49 (m, 4H), 1.43-1.37 (m, 2H), 1.34 (d, *J* = 6.8 Hz, 3H);

¹³C NMR (CDCl₃, 100 MHz) δ 152.8, 146.8, 128.3, 123.5, 64.6, 51.6, 26.2, 24.5, 19.1;

HRMS (ESI): calcd for C₁₃H₁₉N₂O₂ [M+H]⁺: 235.1441. Found 235.1439.

6z Prepared according to the General Procedure A from morpholine (6.0 mmol, 2.0 equiv.), the corresponding potassium aryl acetate (2.0 mmol, 1.0 equiv.), CuI (0.6 mmol, 0.3 equiv.), Zn(OAc)₂ (0.5 mmol, 0.25 equiv) and MnO₂ (10.0 mmol, 5.0 equiv) in 10 mL DMF, 30 °C, 45h. Isolated in 74% yield after purification by silica gel chromatography (2:1 hexane:EtOAc) as a light yellow oil.

¹H NMR (CDCl₃, 700 MHz) δ 7.80 (d, *J* = 8.9 Hz, 1H), 7.09 (d, *J* = 2.9 Hz, 1H), 6.81 (dd, *J* = 8.9 Hz, 2.9 Hz, 1H), 4.19 (q, *J* = 4.7 Hz, 1H), 3.88 (s, 3H), 3.34 (brs, 4H), 2.43-2.31 (m, 4H), 1.88-1.82 (m, 1H), 1.70-1.64 (m, 1H), 1.43 (s, 9H), 1.28-1.15 (m, 2H), 0.87 (t, *J* = 7.5 Hz, 3H);

¹³C NMR (CDCl₃, 176 MHz) δ 162.3, 154.7, 144.3, 139.5, 126.7, 114.3, 111.9, 79.5, 62.3, 55.7, 50.1, 43.3, 33.6, 28.4, 19.0, 14.2;

HRMS (ESI): calcd for C₂₀H₃₂N₃O₅ [M+H]⁺: 394.2336. Found 394.2336.

6aa Prepared according to the General Procedure A from morpholine (6.8 mmol, 2.0 equiv.), the corresponding potassium aryl acetate (3.4 mmol, 1.0 equiv.), CuI (1.02 mmol, 0.3 equiv.), Zn(OAc)₂ (3.4 mmol, 1.0 equiv.), and MnO₂ (17.0 mmol, 5.0 equiv) in 17 mL DMF, rt, 22h. Isolated in 78% yield after purification by silica gel chromatography (2:1 hexane:EtOAc) as a white solid.

¹H NMR (CDCl₃, 700 MHz) δ 7.85 (d, *J* = 8.4 Hz, 1H), 7.82 (d, *J* = 1.8 Hz, 1H), 7.71 (dd, *J* = 8.4 Hz, *J* = 1.8 Hz, 1H), 3.73-3.67 (m, 4H), 3.47 (q, *J* = 6.9 Hz, 1H), 2.52 (brs, 2H), 2.36-2.32 (m, 2H), 1.35 (d, *J* = 6.8 Hz, 3H);

¹³C NMR (CDCl₃, 176 MHz) δ 150.9, 146.9, 131.7, 126.8 (q, *J* = 5.5 Hz), 125.3, 123.9 (q, *J* = 33.8 Hz), 122.0 (q, *J* = 274.3 Hz), 67.0, 64.4, 51.5, 19.5;

¹⁹F NMR (CDCl₃, 470 MHz) δ -59.8;

HRMS (ESI): calcd for C₁₃H₁₆F₃N₂O₃ [M+H]⁺: 305.1108. Found 305.1108.

7a Prepared according to the General Procedure A from NaN₃ (1.0 mmol, 2.0 equiv.), the corresponding potassium aryl acetate (0.5 mmol, 1.0 equiv.), CuI (0.25 mmol, 0.5 equiv.), and MnO₂ (2.5 mmol, 5.0 equiv) in 2.5 mL DMF, rt, 22h. Isolated in 80% yield after purification by silica gel chromatography (99:1 pentane:Et₂O) as a light yellow oil.

¹H NMR (CDCl₃, 500 MHz) δ 7.94 (d, *J* = 8.8 Hz, 1H), 7.67 (d, *J* = 2.4 Hz, 1H), 7.42 (dd, *J* = 8.8 Hz, 2.3 Hz, 1H), 5.26 (q, *J* = 4.1 Hz, 1H), 1.81-1.67 (m, 2H), 1.58-1.44 (m, 2H), 0.97 (t, *J* = 7.2 Hz, 3H);

¹³C NMR (CDCl₃, 126 MHz) δ 146.1, 140.4, 138.4, 128.8, 128.7, 126.3, 60.9, 39.1, 19.6, 13.5;

HRMS (APPI): calcd for C₁₀H₁₂N₂O₂Cl [M-N₂+H]⁺: 227.0582. Found 227.0577.

7b Prepared according to the General Procedure A from NaN₃ (1.0 mmol, 2.0 equiv.), the corresponding potassium aryl acetate (0.5 mmol, 1.0 equiv.), CuI (0.15 mmol, 0.3 equiv.), and MnO₂ (2.5 mmol, 5.0 equiv) in 2.5 mL DMF, 30 °C, 24h. Isolated in 84% yield after purification by silica gel chromatography (15:1 hexane:EtOAc) as a colourless oil.

¹H NMR (CDCl₃, 700 MHz) δ 8.07 (d, *J* = 9.2 Hz, 1H), 7.14 (d, *J* = 3.0 Hz, 1H), 6.89 (dd, *J* = 9.2 Hz, 2.8 Hz, 1H), 5.43 (q, *J* = 4.0 Hz, 1H), 3.92 (s, 3H), 1.81-1.76 (m, 1H), 1.70-1.65 (m, 1H), 1.58-1.47 (m, 2H), 0.97 (t, *J* = 7.5 Hz, 3H);

¹³C NMR (CDCl₃, 176 MHz) δ 163.7, 140.7, 140.0, 127.8, 113.4, 113.2, 61.5, 56.0, 39.2, 19.6, 13.5;

HRMS (APPI): calcd for C₁₁H₁₅N₂O₃ [M-N₂+H]⁺: 223.1077. Found 223.1076.

7d Prepared according to the General Procedure A from NaN₃ (1.0 mmol, 2.0 equiv.), the corresponding potassium aryl acetate (0.5 mmol, 1.0 equiv.), CuI (0.15 mmol, 0.3 equiv.), and MnO₂ (2.5 mmol, 5.0 equiv) in 2.5 mL DMF, 40 °C, 24h. Isolated in 86% yield after purification by silica gel chromatography (40:1 hexane:EtOAc) as a yellow oil.

¹H NMR (CDCl₃, 500 MHz) δ 7.93 (dd, *J* = 8.3 Hz, 1.6 Hz, 1H), 7.72-7.65 (m, 2H), 7.48-7.44 (m, 1H), 5.36 (q, *J* = 6.7 Hz, 1H), 1.59 (d, *J* = 6.6 Hz, 3H);

¹³C NMR (CDCl₃, 126 MHz) δ 148.0, 136.7, 133.8, 128.7, 128.2, 124.7, 56.3, 22.1;

HRMS (APPI): calcd for C₈H₉N₂O₂ [M-N₂+H]⁺: 165.0659. Found 165.0656.

7e Prepared according to the General Procedure A from NaN₃ (1.0 mmol, 2.0 equiv.), the corresponding potassium aryl acetate (0.5 mmol, 1.0 equiv.), CuI (0.15 mmol, 0.3 equiv.), and

MnO₂ (2.5 mmol, 5.0 equiv) in 2.5 mL DMF, rt, 28h. Isolated in 74% yield after purification by silica gel chromatography (40:1 hexane:EtOAc) as a colourless solid.

¹H NMR (CDCl₃, 500 MHz) δ 7.86 (dd, *J* = 8.4 Hz, 2.2 Hz, 1H), 7.73 (d, *J* = 2.2 Hz, 1H), 7.51 (d, *J* = 8.4 Hz, 1H), 5.08 (q, *J* = 6.9 Hz, 1H), 3.96 (s, 3H), 1.50 (d, *J* = 6.8 Hz, 3H);

¹³C NMR (CDCl₃, 126 MHz) δ 156.6, 148.4, 136.9, 127.1, 116.1, 105.6, 56.1, 54.8, 20.2;

HRMS (APPI): calcd for C₉H₁₁N₂O₃ [M-N₂+H]⁺: 195.0764. Found 195.0766.

7f Prepared according to the General Procedure B from NaN₃ (1.0 mmol, 2.0 equiv.), the corresponding aryl acetic acid (0.5 mmol, 1.0 equiv.), CuI (0.15 mmol, 0.3 equiv.), and MnO₂ (2.5 mmol, 5.0 equiv) in 2.5 mL DMF/DCE (1:1), rt, 18h. Isolated in 70% yield after purification by silica gel chromatography (10:1 hexane:EtOAc) as a light yellow oil.

¹H NMR (CDCl₃, 500 MHz) δ 7.91 (d, *J* = 8.3 Hz, 1H), 7.78 (d, *J* = 1.6 Hz, 1H), 7.69 (dd, *J* = 8.3 Hz, 1.6 Hz, 1H), 4.80 (q, *J* = 6.7 Hz, 1H), 1.60 (d, *J* = 6.8 Hz, 3H);

¹³C NMR (CDCl₃, 126 MHz) δ 147.5, 146.8, 130.7, 125.9 (q, *J* = 5.6 Hz), 125.7, 124.3 (q, *J* = 33.5 Hz), 121.8 (d, *J* = 274.3 Hz), 59.6, 21.8;

¹⁹F NMR (CDCl₃, 470 MHz) δ -60.0;

HRMS (EI): calcd for C₉H₇F₃N₄O₂ [M]⁺: 260.0521. Found 260.0520.

7g Prepared according to the General Procedure A from NaN₃ (1.0 mmol, 2.0 equiv.), the corresponding potassium aryl acetate (0.5 mmol, 1.0 equiv.), CuI (0.15 mmol, 0.3 equiv.), and MnO₂ (2.5 mmol, 5.0 equiv) in 2.5 mL DMF, rt, 9h. Isolated in 65% yield after purification by silica gel chromatography (20:1 hexane:EtOAc) as a light yellow oil.

¹H NMR (CDCl₃, 500 MHz) δ 8.24 (d, *J* = 8.7 Hz, 2H), 7.48 (d, *J* = 8.7 Hz, 2H), 4.56 (t, *J* = 7.1 Hz, 1H), 1.88-1.80 (m, 1H), 1.76-1.69 (m, 1H), 1.47-1.29 (m, 2H), 0.95 (t, *J* = 7.3 Hz, 3H);

¹³C NMR (CDCl₃, 126 MHz) δ 147.7, 147.4, 127.7, 124.0, 65.2, 38.5, 19.2, 13.6;

HRMS (APPI): calcd for $C_{10}H_{13}N_2O_2$ $[M-N_2+H]^+$: 193.0972. Found 193.0968.

7h Prepared according to the General Procedure A from NaN_3 (1.0 mmol, 2.0 equiv.), the corresponding potassium aryl acetate (0.5 mmol, 1.0 equiv.), CuI (0.15 mmol, 0.3 equiv.), and MnO_2 (2.5 mmol, 5.0 equiv) in 2.5 mL DMF, rt, 9h. Isolated in 54% yield after purification by silica gel chromatography (20:1 hexane:EtOAc) as a yellow oil.

1H NMR ($CDCl_3$, 500 MHz) δ 8.23 (d, J = 8.8 Hz, 2H), 7.50 (d, J = 8.8 Hz, 2H), 4.75 (q, J = 6.9 Hz, 1H), 1.57 (d, J = 6.9 Hz, 3H);

^{13}C NMR ($CDCl_3$, 126 MHz) δ 148.2, 147.6, 127.2, 124.1, 60.1, 21.7;

HRMS (APPI): calcd for $C_8H_9N_2O_2$ $[M-N_2+H]^+$: 165.0659. Found 165.0656.

The corresponding nitrobenzene **6z** (1.2 mmol), 5% Pd/C (10 wt %), and MeOH (56 mL) were added to 3-neck round bottom flask. After two vacuum/ H_2 cycles to replace air, the mixture was vigorously stirred at room temperature under 1 atm hydrogen pressure (balloon) for 2 h. The mixture was filtered, and evaporated to give the title compound **8** (91% yield) as a yellow oil.

1H NMR ($CDCl_3$, 700 MHz) δ 6.66-6.64 (m, 1H), 6.56-6.54 (m, 2H), 4.47 (s, 2H), 3.73 (s, 3H), 3.44-3.35 (m, 4H), 3.13 (dd, J = 10.4 Hz, 3.8 Hz, 1H), 2.53 (brs, 2H), 2.36-2.32 (m, 2H), 1.91-1.74 (m, 2H), 1.45 (s, 9H), 1.13-1.09 (m, 2H), 0.87 (t, J = 7.4 Hz, 3H);

^{13}C NMR ($CDCl_3$, 176 MHz) δ 154.7, 151.8, 139.5, 126.7, 116.9, 116.7, 112.5, 79.6, 70.4, 55.7, 50.6, 50.5, 44.5, 43.6, 30.4, 28.4, 20.5, 14.3;

HRMS (ESI): calcd for $C_{20}H_{34}N_3O_3$ $[M+H]^+$: 364.2595. Found 364.2594.

The corresponding nitrobenzene **6aa** (1.2 mmol), 5% Pd/C (10 wt %), and MeOH (60 mL) were added to 3-neck round bottom flask. After two vacuum/H₂ cycles to replace air, the mixture was vigorously stirred at room temperature under 1 atm hydrogen pressure (balloon) for 1 h. The mixture was filtered, and evaporated to give the title compound **9** (98% yield) as a yellow oil.

¹H NMR (CDCl₃, 500 MHz) δ 7.33 (d, *J* = 2.0 Hz, 1H), 7.26-7.24 (m, 1H), 7.10 (d, *J* = 8.3 Hz, 1H), 4.11 (s, 2H), 3.68 (t, *J* = 4.7 Hz, 4H), 3.24 (q, *J* = 6.5 Hz, 1H), 2.46-2.32 (m, 4H), 1.30 (d, *J* = 6.7 Hz, 3H);

¹³C NMR (CDCl₃, 126 MHz) δ 143.4, 133.3, 132.1, 125.5 (q, *J* = 5.2 Hz), 125.0 (q, *J* = 272.2 Hz), 117.3, 113.5 (q, *J* = 29.8 Hz), 67.2, 64.3, 51.1, 19.5;

¹⁹F NMR (CDCl₃, 376 MHz) δ -62.5;

HRMS (ESI): calcd for C₁₃H₁₈F₃N₂O [M+H]⁺: 275.1366. Found 275.1365.

Adapted from a literature procedure.⁸ To a solution of **6n** (3 mmol) in anhydrous MeOH (30 mL) was added CuCl (9 mmol). The reaction mixture was stirred at ambient temperature, which KBH₄ (21 mmol) was added to this mixture in portions. After the addition, the reaction mixture was stirred for 10 minutes. MeOH was evaporated under reduced pressure. H₂O was added to the residue and the mixture was extracted with EtOAc (3x). The combined organic phases were washed with brine, dried over Na₂SO₄, filtered, and evaporated to give the title compound **6n'** (97% yield) as a yellow solid.

¹H NMR (CDCl₃, 500 MHz) δ 6.99 (dd, *J* = 8.4 Hz, 2.4 Hz, 1H), 6.88 (d, *J* = 2.1 Hz, 1H), 6.50 (d, *J* = 8.6 Hz, 1H), 4.83 (brs, 2H), 3.72-3.64 (m, 4H), 3.06 (q, *J* = 4.2 Hz, 1H), 2.57 (brs, 2H), 2.37-2.35 (m, 2H), 1.88-1.72 (m, 2H), 1.15-1.02 (m, 2H), 0.86 (t, *J* = 7.4 Hz, 3H);

¹³C NMR (CDCl₃, 126 MHz) δ 144.4, 130.0, 127.5, 126.4, 121.8, 117.1, 71.2, 67.4, 51.7, 30.2, 20.5, 14.3;

HRMS (ESI): calcd for C₁₄H₂₂ClN₂O [M+H]⁺: 269.1415. Found 269.1415.

Adapted from a literature procedure.⁹ To a 4-dram vial charged with a stir bar was sequentially added the corresponding aniline (0.4 mmol) and ethanol (4.8 mL) at 0 °C. To this solution was added conc. H₂SO₄ (0.62 mL) dropwise. This mixture was stirred at 0 °C for 30 min and followed by adding NaNO₂ (1.1 mmol) in portions over 5 min. The reaction was allowed to warm up to rt, then stirred at 80 °C for 2 h. After cooling to rt, sat. NaHCO₃ (20 mL) was added and the mixture was extracted with EtOAc (3 x 50 mL). The combined organic layer was washed with brine, dried over Na₂SO₄, filtered, and concentrated. Isolated in 76% yield after purification by silica gel chromatography (4:1 hexane:EtOAc) as a yellow oil.

¹H NMR (CDCl₃, 700 MHz) δ 7.25-7.21 (m, 3H), 7.11 (dt, *J* = 7.2 Hz, 1.8 Hz, 1H), 3.67 (t, *J* = 4.8 Hz, 4H), 3.18 (q, *J* = 4.9 Hz, 1H), 2.43 (brs, 2H), 2.36-2.33 (m, 2H), 1.86-1.81 (m, 1H), 1.64-1.59 (m, 1H), 1.16-1.04 (m, 2H), 0.85 (t, *J* = 7.4 Hz, 3H);

¹³C NMR (CDCl₃, 176 MHz) δ 143.3, 134.1, 129.3, 128.5, 127.2, 126.8, 70.0, 67.2, 51.1, 34.6, 19.3, 14.1;

HRMS (ESI): calcd for C₁₄H₂₁ClNO [M+H]⁺: 254.1306. Found 254.1306.

Adapted from a literature procedure.¹⁰ To a 1-dram vial charged with a stir bar was sequentially added the corresponding aniline (0.2 mmol) and conc. HCl (0.3 mL) at 0 °C. This mixture was stirred at 0 °C for 30 min followed by adding NaNO₂ (2M in H₂O, 0.6 mmol). After stirring for 1 h, KI (2M in H₂O, 0.8 mmol) was added dropwise and then CH₃CN (600 µL) was added. The solution was vigorously stirred at 0 °C for 30 min. The reaction was allowed to warm to rt, then stirred at 80 °C for 1 h. After cooling to rt, additional EtOAc (50 mL) and water (30 mL) were added, followed by sat. NaHCO₃ (20 mL) and aq. Na₂S₂O₃ (2M, 5 mL). The organic layer was separated and washed with brine, dried over Na₂SO₄, filtered, and concentrated.

Isolated in 62% yield after purification by silica gel chromatography (10:1 hexane:EtOAc) as a yellow oil.

¹H NMR (CDCl₃, 500 MHz) δ 7.73 (d, *J* = 8.4 Hz, 1H), 7.37 (d, *J* = 2.6 Hz, 1H), 6.94 (dd, *J* = 8.4 Hz, 2.6 Hz, 1H), 3.69-3.61 (m, 5H), 2.58-2.56 (m, 2H), 2.38-2.34 (m, 2H), 1.84-1.78 (m, 1H), 1.61-1.54 (m, 1H), 1.20-1.02 (m, 2H), 0.85 (t, *J* = 7.3 Hz, 3H);

¹³C NMR (CDCl₃, 126 MHz) δ 146.2, 140.3, 134.8, 129.1, 128.9, 99.4, 72.1, 67.2, 51.1, 34.5, 18.3, 14.4;

HRMS (ESI): calcd for C₁₄H₂₀ClINO [M+H]⁺: 380.0273. Found 380.0270.

The corresponding nitrobenzene **6x** (3 mmol), 5% Pd/C (10 wt %), and MeOH (140 mL) were added to 3-neck round bottom flask. After two vacuum/H₂ cycles to replace air, the mixture was vigorously stirred at room temperature under 1 atm hydrogen pressure (balloon) for 1 h. The mixture was filtered, and evaporated to give the title compound **6x'** (89% yield) as a yellow oil.

¹H NMR (CDCl₃, 500 MHz) δ 6.99 (d, *J* = 8.3 Hz, 2H), 6.64 (d, *J* = 8.4 Hz, 2H), 3.59 (brs, 2H), 3.25 (brs, 1H), 2.34 (brs, 4H), 1.86-1.73 (m, 2H), 1.59-1.46 (m, 4H), 1.36-1.30 (m, 2H), 1.20-1.08 (m, 2H), 0.84 (t, *J* = 7.4 Hz, 3H);

¹³C NMR (CDCl₃, 126 MHz) δ 145.1, 129.8, 14.6, 112.9, 69.7, 51.1, 34.9, 26.4, 24.7, 20.1, 14.2;

HRMS (ESI): calcd for C₁₅H₂₅N₂ [M+H]⁺: 233.2012. Found 233.2011.

To a 4-dram vial charged with a stir bar was added the corresponding aniline **6x'** (0.2 mmol) and ice (58 mmol). Concentrated H₂SO₄ (6 mmol) was slowly added and the mixture was stirred at rt for 5 minutes. CPME (2.5 mL) was then added to form a biphasic mixture, which was cooled to 5 °C in an ice bath (5 minutes). NaNO₂ (0.39 M in H₂O, 0.22 mmol) was added via syringe over 10 minutes at 5 °C. The vial was sealed with a PTFE cap and pierced with a 22g

needle, and then heated to 80° C for 1 hour. The mixture was cooled to rt and then adjusted to pH >7 with sat. Na₂CO₃. The aqueous phase was extracted with EtOAc (3 x 30 mL), and the combined organic phase was dried over Na₂SO₄, filtered and concentrated, which afforded the desired product **12** in 85% yield as a yellow solid.

¹H NMR (CDCl₃, 600 MHz) δ 7.06 (d, *J* = 8.5 Hz, 2H), 6.74 (d, *J* = 8.5 Hz, 2H), 3.30 (dd, *J* = 10.1 Hz, 4.5 Hz, 1H), 2.38 (brs, 4H), 1.87-1.75 (m, 2H), 1.61-1.51 (m, 5H), 1.37-1.31 (m, 2H), 1.15-1.06 (m, 2H), 0.83 (t, *J* = 7.4 Hz, 3H);

¹³C NMR (CDCl₃, 126 MHz) δ 153.3, 127.7, 127.5, 112.9, 67.4, 48.6, 31.6, 23.2, 21.9, 17.5, 11.5;

HRMS (ESI): calcd for C₁₅H₂₄NO [M+H]⁺: 234.1852. Found 234.1852.

Adapted from a literature procedure.¹¹ 42% HBF₄ (0.6 mL) was added to a solution of the corresponding aniline **6x'** (0.2 mmol) and NaNO₂ (0.24 mmol) in H₂O (0.2 mL) at 0 °C. The mixture was stirred for 1h at and then warmed to rt for 1h, after which methyl acrylate (0.28 mmol), Pd(OAc)₂ (0.01 mmol) and MeOH (0.2 mL) were added to the mixture which was then heated on 50 °C for 1h. After cooling to rt, the mixture was adjusted to pH~9 with Sat. NaHCO₃ and then was extracted with EtOAc (3 x 20 mL). The combined organic layer was washed with brine, dried over Na₂SO₄, filtered, and concentrated. Isolated in 90% yield after purification by silica gel chromatography (15:1 DCM:MeOH) as a light yellow oil.

¹H NMR (CDCl₃, 700 MHz) δ 7.69 (d, *J* = 16.0 Hz, 1H), 7.47 (d, *J* = 8.3 Hz, 2H), 7.24 (d, *J* = 8.3 Hz, 2H), 6.42 (d, *J* = 16.0 Hz, 1H), 3.81 (s, 3H), 3.36 (brs, 1H), 2.37 (brs, 4H), 1.91-1.72 (m, 2H), 1.55 (brs, 4H), 1.35 (brs, 2H), 1.20-1.06 (m, 2H), 0.85 (t, *J* = 7.4 Hz, 3H);

¹³C NMR (CDCl₃, 176 MHz) δ 167.5, 144.7, 133.1, 131.9, 129.3, 127.7, 117.2, 70.1, 51.6, 51.3, 34.6, 26.2, 24.5, 19.7, 14.1;

HRMS (ESI): calcd for C₁₉H₂₈NO₂ [M+H]⁺: 302.2115. Found 302.2115.

IX. Synthesis of Tetrahydroquinoline Core for DUAL946

(2-Nitro- 5-bromo-phenyl)-acetic acid ethyl ester (25 mmol), K₂CO₃ (25 mmol), 18-crown-6 (2 mmol), and potassium iodide (4.75 mmol) were added to a round bottom flask with magnetic stirrer under N₂ atmosphere. Acetonitrile (60 mL) was added to the flask and the mixture was heated at 70 °C for 1h. Chloroacetone (30 mmol) was added to the reaction mixture and stirred at 70 °C for 24 h. The reaction was cooled to room temperature and filtered through a pad of celite. The resulting filtrate was concentrated and purified via flash chromatography (10:1 hexane:EtOAc) to give the compound **A** (91% yield).

Compound **A** (20 mmol), glycol (30 mmol), *p*-TsOH (0.34 mmol), and benzene (80 mL) were added to a 3-neck round bottom flask with magnetic stirrer (Dean–Stark apparatus). The mixture was heated to reflux. After full conversion of the starting material, the reaction was cooled to room temperature. The benzene layer was washed with Sat. NaHCO₃, brine, dried over Na₂SO₄, filtered, and evaporated. Purification via flash chromatography (4:1 hexane:EtOAc) yielded to give the compound **B** (98% yield).

10% KOH was added to a solution of compound B (12 mmol) in MeOH/THF mixture (30 mL, 5:1). The reaction mixture was stirred at room temperature for 4h. After concentrating the reaction mixture, the residue was diluted with water (20 mL) and acidified to pH~3 with 3M HCl. The aqueous solution was extracted with ethyl acetate (40 mL x 3). The combined organic phases were washed with brine, dried over Na₂SO₄, filtered, and evaporated to give the title compound **14a** (98% yield) as a white solid.

¹H NMR (CDCl₃, 500 MHz) δ 7.89 (d, *J* = 8.6 Hz, 1H), 7.69 (d, *J* = 8.0 Hz, 1H), 7.55 (dd, *J* = 8.6 Hz, 2.0 Hz, 1H), 4.43 (dd, *J* = 7.4 Hz, 2.3 Hz, 1H), 3.99-3.83 (m, 4H), 2.78 (dd, *J* = 14.9 Hz, 7.4 Hz, 1H), 2.16 (dd, *J* = 14.9 Hz, 7.4 Hz, 1H), 1.33 (s, 3H);

¹³C NMR (CDCl₃, 126 MHz) δ 177.2, 147.8, 135.9, 134.0, 131.4, 127.8, 126.3, 108.7, 64.7, 64.5, 42.0, 41.3, 24.0;

HRMS (ESI): calcd for C₁₂H₁₃BrNO₄ [M-CO₂-H]⁻: 314.0033. Found 314.0038.

Prepared according to the General Procedure B from NaN₃ (6.12 mmol, 2.0 equiv.), the corresponding aryl acetic acid **14a** (3.06 mmol, 1.0 equiv.), CuI (1.53 mmol, 0.5 equiv.), and MnO₂ (15.3 mmol, 5.0 equiv) in 15.3 mL DMF/DCE (1:1), rt, 36h. Isolated in 92% yield after purification by silica gel chromatography (10:1 hexane:EtOAc) as a colourless oil.

¹H NMR (CDCl₃, 500 MHz) δ 7.82-7.78 (m, 2H), 7.57 (dd, *J* = 8.7 Hz, 2.1 Hz, 1H), 5.44 (dd, *J* = 5.3 Hz, 2.2 Hz, 1H), 3.99-3.92 (m, 4H), 2.16-2.15 (m, 2H), 1.41 (s, 3H);

¹³C NMR (CDCl₃, 126 MHz) δ 146.6, 138.4, 132.2, 131.9, 128.5, 126.0, 108.1, 64.6, 64.5, 56.8, 45.2, 24.4;

HRMS (APPI): calcd for C₁₂H₁₃N₂O₄Br [M-N₂+H]⁺: 329.0131. Found 329.0126.

Adapted from a literature procedure.¹² Ph₃P (4.5 mmol) was added to a solution of **14b** (3 mmol) in dry MeOH (30 mL). The reaction mixture was refluxed at 80 °C for 3d. After all the starting material and intermediate had disappeared (monitored by ¹H NMR), the reaction mixture was cooled to room temperature and the solvent was removed under reduced press. H₂O was added to the residue and the mixture was extracted with DCM (3x). The combined organic phases were washed with brine, dried over Na₂SO₄, filtered, and evaporated. Chromatography on silica gel (10:1 DCM:MeOH) gave the title compound **14c** (97% yield) as white solid.

¹H NMR (CDCl₃, 500 MHz) δ 8.12 (d, *J* = 2.5 Hz, 1H), 7.67 (d, *J* = 8.5 Hz, 1H), 7.48 (dd, *J* = 8.5 Hz, 2.5 Hz, 1H), 4.74 (dd, *J* = 9.5 Hz, 2.5 Hz, 1H), 4.01-3.92 (m, 4H), 2.08-1.90 (m, 4H), 1.42 (s, 3H);

¹³C NMR (CDCl₃, 126 MHz) δ 147.4, 143.5, 132.4, 130.7, 128.0, 125.5, 109.6, 64.8, 64.2, 47.1, 46.4, 23.8;

HRMS (ESI): calcd for C₁₂H₁₆BrN₂O₄ [M+H]⁺: 331.0288. Found 331.0289.

Et₃N (1.5 mmol) and isopropyl chloroformate (1M in toluene, 1.5 mmol) were added successively to a solution of **14c** (1.5 mmol) in dry DCM (10 mL) at 0 °C. The reaction mixture was stirred at 0 °C. After all the starting material had disappeared (monitored by ¹H NMR), H₂O was added to the mixture. The mixture was extracted with DCM (3x). The combined organic phases were washed with brine, dried over Na₂SO₄, filtered, and evaporated. Chromatography on silica gel (5:1 hexane:EtOAc) gave the title compound **14d** (91% yield) as white solid.

¹H NMR (CDCl₃, 700 MHz) δ 7.75-7.72 (m, 2H), 7.47 (dd, *J* = 8.4 Hz, 1.4 Hz, 1H), 6.14 (s, 1H), 5.29 (s, 1H), 4.78 (s, 1H), 4.03-3.86 (m, 4H), 2.25 (dd, *J* = 7.7 Hz, 3.5 Hz, 1H), 1.94 (s, 1H), 1.38 (s, 3H), 1.18-0.70 (m, 6H);

¹³C NMR (CDCl₃, 176 MHz) δ 155.7, 147.0, 141.5, 131.4, 130.8, 128.2, 126.3, 109.1, 68.5, 65.0, 64.0, 48.9, 43.3, 23.4, 21.9;

HRMS (ESI): calcd for C₁₆H₂₁BrN₂O₆Na [M+Na]⁺: 439.0475. Found 439.0476.

To a solution of **14d** (1 mmol) in THF/MeOH(2:1, 9 mL) was added HCl (3 M, 4 mL). The reaction mixture was stirred at ambient temperature for 8 h. Saturated NaHCO₃ was added to neutralize HCl solution. The aqueous phase was extracted with DCM (3x). The combined organic phases were washed with water and brine, dried over Na₂SO₄, filtered, and evaporated. Chromatography on silica gel (4:1 hexane:EtOAc) gave the title compound **14e** (98% yield) as white solid.

¹H NMR (CDCl₃, 700 MHz) δ 7.81 (d, *J* = 1.4 Hz, 2H), 7.54-7.52 (m, 1H), 5.95 (s, 1H), 5.59 (s, 1H), 4.81 (t, *J* = 5.6 Hz, 1H), 3.10-2.97 (m, 2H), 2.17 (s, 3H), 1.24-1.17 (m, 6H);

^{13}C NMR (CDCl_3 , 176 MHz) δ 155.3, 146.8, 139.5, 132.4, 131.5, 131.4, 128.7, 126.6, 68.9, 48.1, 47.8, 30.2, 22.0;

HRMS (ESI): calcd for $\text{C}_{14}\text{H}_{17}\text{BrN}_2\text{O}_5\text{Na}$ $[\text{M}+\text{Na}]^+$: 395.0213. Found 395.0211.

To a solution of **14e** (0.2 mmol) in anhydrous MeOH (2 mL) was added CuCl (0.6 mmol). The reaction mixture was stirred at $-10\text{ }^\circ\text{C}$, which KBH_4 (1.4 mmol) was added to this mixture in portions. After the addition, the reaction mixture was stirred for 20 minutes at $-10\text{ }^\circ\text{C}$. MeOH was evaporated under reduced pressure. H_2O was added to the residue and the mixture was extracted with DCM (3x). The combined organic phases were washed with brine, dried over Na_2SO_4 , filtered, and evaporated. Chromatography on silica gel (5:1 hexane: EtOAc) gave the title compound **14f** (71% yield, *syn*, 6:1 dr) as white solid. Spectroscopic data agreed with that reported.¹³

^1H NMR (CDCl_3 , 700 MHz) δ 7.26 (s, 1H), 7.08 (dd, $J = 8.4\text{ Hz}$, 1.5 Hz , 1H), 6.35 (d, $J = 8.4\text{ Hz}$, 1H), 4.99-4.97 (m, 2H), 4.67 (d, $J = 2.8\text{ Hz}$, 1H), 3.72 (s, 1H), 3.56-3.54 (m, 1H), 2.28-2.26 (m, 1H), 1.44 (q, $J = 11.9\text{ Hz}$, 1H), 1.31-1.26 (m, 6H), 1.21 (d, $J = 6.3\text{ Hz}$, 3H);

^{13}C NMR (CDCl_3 , 176 MHz) δ 156.2, 144.0, 130.9, 129.4, 123.7, 115.6, 109.0, 68.5, 47.7, 46.7, 37.9, 22.2, 22.1.

X. References

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XI ¹H and ¹³C Spectra

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **v700, Oct 25 2018**

Pulse Sequence: **PRESAT**

Sweep Width(Hz): **6389.26**

Digital Res.(Hz/pt): **0.13**

Acquisition Time(s): **5**

Hz per mm(Hz/mm): **27.96**

Relaxation Delay(s): **0.1**

Completed Scans **8**

DK-7-155-C

699.762 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe

2a

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **v700, Oct 25 2018**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **48076.9**
Digital Res.(Hz/pt): **0.37**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **159.99**

Relaxation Delay(s): **1**
Completed Scans **184**

DK-7-155-C

175.975 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **v700, Jul 21 2018**
Pulse Sequence: **PRESAT**

Sweep Width(Hz): **8389.26**
Digital Res.(Hz/pt): **0.13**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **27.48**

Relaxation Delay(s): **0.1**
Completed Scans **8**

DK-5-203-A

699.762 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe

2b

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **v700, Jul 21 2018**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **48076.9**
Digital Res.(Hz/pt): **0.37**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **158.04**

Relaxation Delay(s): **1**
Completed Scans: **512**

DK-5-203-A
175.975 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **u500, Jan 24 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **6009.62**
Digital Res.(Hz/pt): **0.09**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **20**

Relaxation Delay(s): **0.1**
Completed Scans **16**

OB-1-69

499.797 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 26.1 C -> actual temp = 27.0 C, autotx probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **u500, Jan 24 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **116.55**

Relaxation Delay(s): **1**
Completed Scans **548**

OB-1-69

125.688 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 26.1 C -> actual temp = 27.0 C, autoxdb probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **u500, Jun 3 2019**

Sweep Width(Hz): **6009.62**

Acquisition Time(s): **5**

Relaxation Delay(s): **0.1**

Pulse Sequence: **PRESAT**

Digital Res.(Hz/pt): **0.09**

Hz per mm(Hz/mm): **20.72**

Completed Scans **8**

DK-10-169-A

499.787 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **u500, Jun 1 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **117.84**

Relaxation Delay(s): **1**
Completed Scans: **4**

DK-10-169-A

125.685 MHz $^{13}\text{C}\{^1\text{H}\}$ 1D in cdcl_3 (ref. to CDCl_3 @ 77.06 ppm) temp 27.7 C -> actual temp = 27.0 C, coldddual probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **v700, Jun 4 2019**
Pulse Sequence: **PRESAT**

Sweep Width(Hz): **8389.26**
Digital Res.(Hz/pt): **0.13**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **29.39**

Relaxation Delay(s): **0.1**
Completed Scans **8**

DK-10-177

699.762 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: v700, Jun 4 2019
Pulse Sequence: s2pul

Sweep Width(Hz): 36764.7
Digital Res.(Hz/pt): 0.28

Acquisition Time(s): 1
Hz per mm(Hz/mm): 153.19

Relaxation Delay(s): 1
Completed Scans 156

DK-10-177

175.971 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **u500, Jun 3 2019**
Pulse Sequence: **PRESAT**

Sweep Width(Hz): **6009.62**
Digital Res.(Hz/pt): **0.09**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **20.7**

Relaxation Delay(s): **0.1**
Completed Scans **8**

DK-10-169-C

499.787 MHz H1 1D in cdcl3 (ref. to CDCI3 @ 7.26 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe

DK-10-169-C

125.685 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe

Department of Chemistry, University of Alberta

Recorded on: **mr400, Feb 25 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **4807.69**
Digital Res.(Hz/pt): **0.07**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **15.58**

Relaxation Delay(s): **0.1**
Completed Scans **16**

DK-9-153

399.978 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 25.9 C -> actual temp = 27.0 C, onenmr probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **mr400, Feb 25 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **27173.9**
Digital Res.(Hz/pt): **0.21**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **92.15**

Relaxation Delay(s): **1**
Completed Scans **340**

DK-9-153

100.586 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 25.9 C -> actual temp = 27.0 C, onenmr probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **i400, Oct 27 2018**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **4801.92**
Digital Res.(Hz/pt): **0.07**

Acquisition Time(s): **4.998**
Hz per mm(Hz/mm): **15.43**

Relaxation Delay(s): **0.1**
Completed Scans **16**

DK-7-159-A

399.794 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 26.5 C -> actual temp = 27.0 C, autotdb probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **v700, Oct 26 2018**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **48076.9**
Digital Res.(Hz/pt): **0.37**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **161.04**

Relaxation Delay(s): **1**
Completed Scans **256**

DK-7-159-A
175.975 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **mr400, Nov 2 2018**

Sweep Width(Hz): **4807.69**

Acquisition Time(s): **5**

Relaxation Delay(s): **0.1**

Pulse Sequence: **s2pul**

Digital Res.(Hz/pt): **0.07**

Hz per mm(Hz/mm): **15.84**

Completed Scans **12**

DK-7-191-A

399.978 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 25.9 C -> actual temp = 27.0 C, onenmr probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **mr400, Nov 2 2018**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **27173.9**
Digital Res.(Hz/pt): **0.21**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **91.21**

Relaxation Delay(s): **1**
Completed Scans **220**

DK-7-191-A

100.586 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 25.9 C -> actual temp = 27.0 C, onenmr probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **mr400, Nov 2 2018**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **4807.69**
Digital Res.(Hz/pt): **0.07**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **15.37**

Relaxation Delay(s): **0.1**
Completed Scans **12**

DK-7-177-B

399.978 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 25.9 C -> actual temp = 27.0 C, onenmr probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **i400, Nov 2 2018**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **26954.2**
Digital Res.(Hz/pt): **0.21**

Acquisition Time(s): **1.002**
Hz per mm(Hz/mm): **88.96**

Relaxation Delay(s): **1**
Completed Scans **300**

DK-7-177-B
100.539 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 26.5 C -> actual temp = 27.0 C, autotdb probe

Department of Chemistry, University of Alberta

Recorded on: **mr400, Oct 27 2018**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **4807.69**
Digital Res.(Hz/pt): **0.07**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **15.57**

Relaxation Delay(s): **0.1**
Completed Scans **10**

DK-7-159-C

399.978 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) Temp 25.9 C -> actual temp = 27.0 C, onenmr probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **mr400, Oct 27 2018**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **27173.9**
Digital Res.(Hz/pt): **0.21**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **92.43**

Relaxation Delay(s): **1**
Completed Scans **300**

DK-7-159-C

100.586 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 25.9 C -> actual temp = 27.0 C, onenmr probe

Department of Chemistry, University of Alberta

Recorded on: **u500, Feb 16 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **6009.62**
Digital Res.(Hz/pt): **0.09**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **19.66**

Relaxation Delay(s): **0.1**
Completed Scans **16**

DK-9-129

499.797 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 26.1 C -> actual temp = 27.0 C, autotx probe

5f

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **u500, Feb 16 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **114.11**

Relaxation Delay(s): **1**
Completed Scans **300**

DK-9-129

125.688 MHz $^{13}\text{C}\{^1\text{H}\}$ 1D in cdcl_3 (ref. to CDCl_3 @ 77.06 ppm) temp 26.1 C -> actual temp = 27.0 C, autoxdb probe

5f

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **v700, Oct 25 2018**
Pulse Sequence: **PRESAT**

Sweep Width(Hz): **8389.26**
Digital Res.(Hz/pt): **0.13**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **28.37**

Relaxation Delay(s): **0.1**
Completed Scans: **8**

DK-7-153-A

699.762 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe

5g

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **v700, Oct 25 2018**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **48076.9**
Digital Res.(Hz/pt): **0.37**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **159.08**

Relaxation Delay(s): **1**
Completed Scans **512**

DK-7-153-A
175.975 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe

5g

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **i600, Oct 25 2018**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **7183.91**
Digital Res.(Hz/pt): **0.11**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **23.93**

Relaxation Delay(s): **0.1**
Completed Scans **16**

DK-7-153-B

599.926 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 26.2 C -> actual temp = 27.0 C, autoxid probe

5h

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **v700, Oct 24 2018**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **48076.9**
Digital Res.(Hz/pt): **0.37**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **159.73**

Relaxation Delay(s): **1**
Completed Scans **256**

DK-7-153-B
175.975 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **u500, Jan 22 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **6009.62**
Digital Res.(Hz/pt): **0.09**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **19.4**

Relaxation Delay(s): **0.1**
Completed Scans **8**

DK-8-137

499.797 MHz ¹H 1D in cdcl₃ (ref. to CDC13 @ 7.26 ppm) temp 26.1 C -> actual temp = 27.0 C, autotdb probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **u500, Jan 22 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **119.48**

Relaxation Delay(s): **1**
Completed Scans **116**

DK-8-137

125.688 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 26.1 C -> actual temp = 27.0 C, autotdb probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **v700, Mar 20 2019**
Pulse Sequence: **PRESAT**

Sweep Width(Hz): **8389.26**
Digital Res.(Hz/pt): **0.13**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **26.93**

Relaxation Delay(s): **0.1**
Completed Scans **8**

DK-10-47

699.762 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe

5j

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **v700, Mar 20 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **36764.7**
Digital Res.(Hz/pt): **0.28**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **153.19**

Relaxation Delay(s): **1**
Completed Scans: **176**

DK-10-47

175.971 MHz $^{13}\text{C}\{^1\text{H}\}$ 1D in cdCl_3 (ref. to CDCl_3 @ 77.06 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe

5j

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **u500, Mar 2 2019**
Pulse Sequence: **PRESAT**

Sweep Width(Hz): **6009.62**
Digital Res.(Hz/pt): **0.09**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **19.39**

Relaxation Delay(s): **0.1**
Completed Scans **8**

DK-9-171

499.787 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe

Department of Chemistry, University of Alberta

Recorded on: **u500, Mar 2 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **119.4**

Relaxation Delay(s): **1**
Completed Scans **20**

DK-9-171

125.685 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.7 C -> actual temp = 27.0 C, coldlual probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **v700, Mar 22 2019**
Pulse Sequence: **PRESAT**

Sweep Width(Hz): **8389.26**
Digital Res.(Hz/pt): **0.13**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **27.42**

Relaxation Delay(s): **0.1**
Completed Scans: **8**

DK-10-53

699.762 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **v700, Mar 22 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **36764.7**
Digital Res.(Hz/pt): **0.28**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **153.19**

Relaxation Delay(s): **1**
Completed Scans **12**

DK-10-53

175.971 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **v700, Nov 1 2018**
Pulse Sequence: **PRESAT**

Sweep Width(Hz): **8389.26**
Digital Res.(Hz/pt): **0.13**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **31.22**

Relaxation Delay(s): **0.1**
Completed Scans **8**

DK-7-175

699.762 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **v700, Nov 1 2018**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **48076.9**
Digital Res.(Hz/pt): **0.37**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **160.39**

Relaxation Delay(s): **1**
Completed Scans **176**

DK-7-175
175.975 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **u500, Feb 7 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **6009.62**
Digital Res.(Hz/pt): **0.09**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **19.66**

Relaxation Delay(s): **0.1**
Completed Scans: **16**

DK-9-89

499.797 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 26.1 C -> actual temp = 27.0 C, autotdb probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **u500, Feb 6 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **115.45**

Relaxation Delay(s): **1**
Completed Scans **192**

DK-9-89

125.688 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 26.1 C -> actual temp = 27.0 C, autotdb probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **mr400, Oct 27 2018**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **4807.69**
Digital Res.(Hz/pt): **0.07**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **15.61**

Relaxation Delay(s): **0.1**
Completed Scans **10**

DK-7-159-B

399.978 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 25.9 C -> actual temp = 27.0 C, onenmr probe

5o

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **mr400, Oct 27 2018**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **27173.9**
Digital Res.(Hz/pt): **0.21**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **92.15**

Relaxation Delay(s): **1**
Completed Scans **300**

DK-7-159-B
100.586 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 25.9 C -> actual temp = 27.0 C, onenmr probe

5o

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **mr400, Nov 2 2018**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **4807.69**
Digital Res.(Hz/pt): **0.07**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **15.89**

Relaxation Delay(s): **0.1**
Completed Scans **12**

DK-7-177-A

399.978 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 25.9 C -> actual temp = 27.0 C, onenmr probe

5p

S-92

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **mr400, Nov 2 2018**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **27173.9**
Digital Res.(Hz/pt): **0.21**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **91.64**

Relaxation Delay(s): **1**
Completed Scans: **4**

DK-7-177-A

100.586 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 25.9 C -> actual temp = 27.0 C, onenmr probe

5p

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **u500, Mar 9 2019**
Pulse Sequence: **PRESAT**

Sweep Width(Hz): **6009.62**
Digital Res.(Hz/pt): **0.09**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **19.65**

Relaxation Delay(s): **0.1**
Completed Scans **8**

DK-9-181

499.787 MHz H1 1D in cdcl3 (ref. to CDC13 @ 7.26 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe

S-94

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **u500, Mar 9 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **117.66**

Relaxation Delay(s): **1**
Completed Scans **464**

DK-9-181

125.685 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **mr400, Feb 25 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **4807.69**
Digital Res.(Hz/pt): **0.07**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **15.27**

Relaxation Delay(s): **0.1**
Completed Scans **16**

DK-9-143

399.978 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm)temp 25.9 C -> actual temp = 27.0 C, onenmr probe

5s

S-96

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **mr400, Feb 25 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **27173.9**
Digital Res.(Hz/pt): **0.21**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **93.57**

Relaxation Delay(s): **1**
Completed Scans **284**

DK-9-143

100.586 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 25.9 C -> actual temp = 27.0 C, onenmr probe

5s

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **mr400, Nov 3 2018**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **4807.69**
Digital Res.(Hz/pt): **0.07**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **15.69**

Relaxation Delay(s): **0.1**
Completed Scans **16**

DK-7-191-B

399.978 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 25.9 C -> actual temp = 27.0 C, onenmr probe

5t

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **mr400, Nov 3 2018**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **27173.9**
Digital Res.(Hz/pt): **0.21**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **91.14**

Relaxation Delay(s): **1**
Completed Scans **396**

DK-7-191-B

100.586 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 25.9 C -> actual temp = 27.0 C, onenmr probe

5t

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **u500, Feb 28 2019**
Pulse Sequence: **PRESAT**

Sweep Width(Hz): **6009.62**
Digital Res.(Hz/pt): **0.09**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **19.26**

Relaxation Delay(s): **0.1**
Completed Scans **8**

DK-9-151

499.787 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe

5u

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **u500, Feb 28 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **119.58**

Relaxation Delay(s): **1**
Completed Scans: **8**

DK-9-151

125.685 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe

S-101

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **u500, Jan 25 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **6009.62**
Digital Res.(Hz/pt): **0.09**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **19.59**

Relaxation Delay(s): **0.1**
Completed Scans **16**

DK-9-49

499.797 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 26.1 C -> actual temp = 27.0 C, autotdb probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **u500, Jan 25 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **114.2**

Relaxation Delay(s): **1**
Completed Scans **632**

DK-9-49

125.688 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 26.1 C -> actual temp = 27.0 C, autotdb probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **mr400, Nov 10 2018**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **4807.69**
Digital Res.(Hz/pt): **0.07**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **15.71**

Relaxation Delay(s): **0.1**
Completed Scans **16**

DK-7-203

399.978 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 25.9 C -> actual temp = 27.0 C, onenmr probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **mr400, Nov 10 2018**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **27173.9**
Digital Res.(Hz/pt): **0.21**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **97.47**

Relaxation Delay(s): **1**
Completed Scans **4844**

DK-7-203

100.586 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 25.9 C -> actual temp = 27.0 C, onenmr probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **v700, Dec 20 2018**
Pulse Sequence: **PRESAT**

Sweep Width(Hz): **8389.26**
Digital Res.(Hz/pt): **0.13**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **27.35**

Relaxation Delay(s): **0.1**
Completed Scans: **8**

DK-8-157-B

699.762 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **v700, Dec 20 2018**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **36764.7**
Digital Res.(Hz/pt): **0.28**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **153.08**

Relaxation Delay(s): **1**
Completed Scans **936**

DK-8-157-B
175.971 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **ibd5, Dec 15 2018**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **6000.6**
Digital Res.(Hz/pt): **0.09**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **19.39**

Relaxation Delay(s): **0.1**
Completed Scans **16**

DK-8-147-A

498.118 MHz H1 1D in cdcl3 (ref. to CDC13 @ 7.26 ppm) temp 26.9 C -> actual temp = 27.0 C, autotx probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **v700, Dec 15 2018**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **36764.7**
Digital Res.(Hz/pt): **0.28**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **153.19**

Relaxation Delay(s): **1**
Completed Scans **5000**

DK-8-147-A

175.971 MHz C13{H1} 1D in cdcl3 (ref. to CDC13 @ 77.06 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **v700, Jan 30 2019**
Pulse Sequence: **PRESAT**

Sweep Width(Hz): **8389.26**
Digital Res.(Hz/pt): **0.13**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **30.28**

Relaxation Delay(s): **0.1**
Completed Scans **8**

DK-9-69

699.762 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe

5z

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **v700, Jan 30 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **36764.7**
Digital Res.(Hz/pt): **0.28**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **152.88**

Relaxation Delay(s): **1**
Completed Scans **256**

DK-9-69

175.971 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **u500, Mar 18 2019**
Pulse Sequence: **PRESAT**

Sweep Width(Hz): **6009.62**
Digital Res.(Hz/pt): **0.09**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **21.33**

Relaxation Delay(s): **0.1**
Completed Scans **8**

DK-10-29-A

499.787 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **u500, Mar 18 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **117.29**

Relaxation Delay(s): **1**
Completed Scans: **128**

DK-10-29-A

125.685 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **v700, Oct 25 2018**
Pulse Sequence: **PRESAT**

Sweep Width(Hz): **8389.26**
Digital Res.(Hz/pt): **0.13**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **27.25**

Relaxation Delay(s): **0.1**
Completed Scans **8**

DK-7-155-A

699.762 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe

6a

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **v700, Oct 25 2018**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **48076.9**
Digital Res.(Hz/pt): **0.37**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **159.08**

Relaxation Delay(s): **1**
Completed Scans **56**

DK-7-155-A

175.975 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **v700, Aug 24 2018**
Pulse Sequence: **PRESAT**

Sweep Width(Hz): **8389.26**
Digital Res.(Hz/pt): **0.13**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **27.91**

Relaxation Delay(s): **0.1**
Completed Scans **8**

DK-6-115

699.762 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe

6b

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **v700, Aug 18 2018**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **48076.9**
Digital Res.(Hz/pt): **0.37**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **154.25**

Relaxation Delay(s): **1**
Completed Scans **256**

DK-6-115

175.975 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **u500, Feb 8 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **6009.62**
Digital Res.(Hz/pt): **0.09**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **19.4**

Relaxation Delay(s): **0.1**
Completed Scans **16**

DK-9-87-A

499.797 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 26.1 C -> actual temp = 27.0 C, autotdb probe

6c

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **u500, Feb 8 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **116.51**

Relaxation Delay(s): **1**
Completed Scans **396**

DK-9-87-A

125.688 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 26.1 C -> actual temp = 27.0 C, autotdb probe

6c

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **v700, Apr 11 2019**
Pulse Sequence: **PRESAT**

Sweep Width(Hz): **8389.26**
Digital Res.(Hz/pt): **0.13**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **28.98**

Relaxation Delay(s): **0.1**
Completed Scans **8**

DK-10-97

699.762 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe

S-120

Relaxation Delay(s): 1
Completed Scans 60

175.971 MHz C13{H1} 1D in cdcl3 (ref. to CDC13 @ 77.06 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **v700, Apr 10 2019**
Pulse Sequence: **PRESAT**

Sweep Width(Hz): **6389.26**
Digital Res.(Hz/pt): **0.13**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **27.73**

Relaxation Delay(s): **0.1**
Completed Scans **4**

DK-10-95

699.762 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe

6e

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **v700, Apr 10 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **40322.6**
Digital Res.(Hz/pt): **0.31**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **153.19**

Relaxation Delay(s): **1**
Completed Scans: **136**

DK-10-95

175.973 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe

6e

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **v700, Jan 31 2019**
Pulse Sequence: **PRESAT**

Sweep Width(Hz): **8389.26**
Digital Res.(Hz/pt): **0.13**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **24.88**

Relaxation Delay(s): **0.1**
Completed Scans **8**

DK-9-57-B

699.762 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe

S-124

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **v700, Jan 31 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **36764.7**
Digital Res.(Hz/pt): **0.28**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **153.19**

Relaxation Delay(s): **1**
Completed Scans **176**

DK-9-57-B

175.971 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **v700, Apr 4 2019**
Pulse Sequence: **PRESAT**

Sweep Width(Hz): **6389.26**
Digital Res.(Hz/pt): **0.13**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **27.73**

Relaxation Delay(s): **0.1**
Completed Scans **8**

DK-10-77

699.762 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **v700, Apr 4 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **36764.7**
Digital Res.(Hz/pt): **0.28**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **153.19**

Relaxation Delay(s): **1**
Completed Scans **32**

DK-10-77

175.971 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **u500, Feb 7 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **6009.62**
Digital Res.(Hz/pt): **0.09**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **19.69**

Relaxation Delay(s): **0.1**
Completed Scans **16**

DK-9-87-B

499.797 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 26.1 C -> actual temp = 27.0 C, autotdb probe

6h

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **u500, Feb 7 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **116.15**

Relaxation Delay(s): **1**
Completed Scans: **148**

DK-9-87-B

125.688 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 26.1 C -> actual temp = 27.0 C, autotx probe

6h

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **ibd5, Nov 28 2018**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **6000.6**
Digital Res.(Hz/pt): **0.09**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **20.09**

Relaxation Delay(s): **0.1**
Completed Scans: **16**

DK-8-71-A

498.118 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 26.9 C -> actual temp = 27.0 C, autotdb probe

6i

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **ibd5, Nov 26 2018**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **33826.6**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **0.998**
Hz per mm(Hz/mm): **115.24**

Relaxation Delay(s): **1**
Completed Scans: **524**

DK-8-71-A
125.266 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 26.9 C -> actual temp = 27.0 C, autotdb probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **u500, Jan 23 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **6009.62**
Digital Res.(Hz/pt): **0.09**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **19.91**

Relaxation Delay(s): **0.1**
Completed Scans **16**

DK-9-61

499.797 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 26.1 C -> actual temp = 27.0 C, autotune probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: u500, Jan 23 2019
Pulse Sequence: s2pul

Sweep Width(Hz): 33783.8
Digital Res.(Hz/pt): 0.26

Acquisition Time(s): 1
Hz per mm(Hz/mm): 114.47

Relaxation Delay(s): 1
Completed Scans 528

DK-9-61

125.688 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 26.1 C -> actual temp = 27.0 C, autotdb probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **v700, Feb 4 2019**
Pulse Sequence: **PRESAT**

Sweep Width(Hz): **6369.26**
Digital Res.(Hz/pt): **0.13**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **27.57**

Relaxation Delay(s): **0.1**
Completed Scans **8**

DK-9-95

699.762 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **v700, Feb 4 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **36764.7**
Digital Res.(Hz/pt): **0.28**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **153.19**

Relaxation Delay(s): **1**
Completed Scans **32**

DK-9-95

175.971 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **u500, Jan 12 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **6009.62**
Digital Res.(Hz/pt): **0.09**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **19.58**

Relaxation Delay(s): **0.1**
Completed Scans **16**

DK-8-193

499.797 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 26.1 C -> actual temp = 27.0 C, autotdb probe

Department of Chemistry, University of Alberta

Recorded on: **u500, Jan 12 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **115.63**

Relaxation Delay(s): **1**
Completed Scans **1492**

DK-8-193

125.688 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 26.1 C -> actual temp = 27.0 C, autotdb probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **ibds, Nov 17 2018**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **6000.6**
Digital Res.(Hz/pt): **0.09**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **19.65**

Relaxation Delay(s): **0.1**
Completed Scans **16**

DK-8-61

498.118 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 26.9 C -> actual temp = 27.0 C, autotdb probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **ibd5, Nov 17 2018**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **33826.6**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **0.998**
Hz per mm(Hz/mm): **115.07**

Relaxation Delay(s): **1**
Completed Scans **308**

DK-8-61

125.266 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 26.9 C -> actual temp = 27.0 C, autotx probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **mr400, Mar 15 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **4807.69**
Digital Res.(Hz/pt): **0.07**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **15.57**

Relaxation Delay(s): **0.1**
Completed Scans **16**

OB-1-137

399.978 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 25.9 C -> actual temp = 27.0 C, onenmr probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **ibd5, Mar 15 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **33826.6**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **0.998**
Hz per mm(Hz/mm): **110.45**

Relaxation Delay(s): **1**
Completed Scans **400**

OB-1-137

125.266 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 26.9 C -> actual temp = 27.0 C, autotdb probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **v700, Dec 1 2018**
Pulse Sequence: **PRESAT**

Sweep Width(Hz): **8389.26**
Digital Res.(Hz/pt): **0.13**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **27.82**

Relaxation Delay(s): **0.1**
Completed Scans **8**

DK-8-109-A

699.762 MHz ¹H 1D in cdcl₃ (ref. to CDCl₃ @ 7.26 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe

S-142

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **v700, Nov 30 2018**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **48076.9**
Digital Res.(Hz/pt): **0.37**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **159.47**

Relaxation Delay(s): **1**
Completed Scans **256**

DK-8-109-A
175.975 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **ibd5, Feb 13 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **6000.6**
Digital Res.(Hz/pt): **0.09**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **20.07**

Relaxation Delay(s): **0.1**
Completed Scans **4**

OB-1-103

498.118 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 26.9 C -> actual temp = 27.0 C, autoxdb probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **ibd5, Feb 13 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **33826.6**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **0.998**
Hz per mm(Hz/mm): **114.77**

Relaxation Delay(s): **1**
Completed Scans **232**

OB-1-103

125.266 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 26.9 C -> actual temp = 27.0 C, autotdb probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **v700, Dec 18 2018**
Pulse Sequence: **PRESAT**

Sweep Width(Hz): **8389.26**
Digital Res.(Hz/pt): **0.13**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **27.07**

Relaxation Delay(s): **0.1**
Completed Scans **8**

DK-8-155

699.762 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **v700, Dec 18 2018**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **36764.7**
Digital Res.(Hz/pt): **0.28**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **153.19**

Relaxation Delay(s): **1**
Completed Scans **3000**

DK-8-155

175.971 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe

Department of Chemistry, University of Alberta

Recorded on: **u500, Mar 19 2019**
Pulse Sequence: **PRESAT**

Sweep Width(Hz): **6009.62**
Digital Res.(Hz/pt): **0.09**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **19.9**

Relaxation Delay(s): **0.1**
Completed Scans **8**

DK-10-35

499.787 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe

S-148

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **u500, Mar 19 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **118.21**

Relaxation Delay(s): **1**
Completed Scans **256**

DK-10-35

125.685 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) emp 27.7 C -> actual temp = 27.0 C, cold dual probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **v700, Nov 16 2018**
Pulse Sequence: **PRESAT**

Sweep Width(Hz): **8389.26**
Digital Res.(Hz/pt): **0.13**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **29.51**

Relaxation Delay(s): **0.1**
Completed Scans **8**

DK-8-59-A

699.762 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe

S-150

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **v700, Nov 16 2018**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **48076.9**
Digital Res.(Hz/pt): **0.37**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **162.73**

Relaxation Delay(s): **1**
Completed Scans **256**

DK-8-59-A

175.975 MHz $^{13}\text{C}\{^1\text{H}\}$ 1D in cdCl_3 (ref. to CDCl_3 @ 77.06 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe

6s

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **u500, Mar 5 2019**
Pulse Sequence: **PRESAT**

Sweep Width(Hz): **6009.62**
Digital Res.(Hz/pt): **0.09**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **19.35**

Relaxation Delay(s): **0.1**
Completed Scans **8**

DK-9-169

499.787 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **u500, Mar 5 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **117.75**

Relaxation Delay(s): **1**
Completed Scans **128**

DK-9-169

125.685 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **mr400, Dec 7 2018**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **4807.69**
Digital Res.(Hz/pt): **0.07**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **16.11**

Relaxation Delay(s): **0.1**
Completed Scans **16**

DK-8-127-A

399.978 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 25.9 C -> actual temp = 27.0 C, onenmr probe

S-154

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **mr400, Dec 7 2018**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **27173.9**
Digital Res.(Hz/pt): **0.21**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **92.07**

Relaxation Delay(s): **1**
Completed Scans **300**

DK-8-127-A

100.586 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 25.9 C -> actual temp = 27.0 C, onenmr probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **u500, Jan 21 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **6009.62**
Digital Res.(Hz/pt): **0.09**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **20.2**

Relaxation Delay(s): **0.1**
Completed Scans **16**

DK-9-27

499.797 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 26.1 C -> actual temp = 27.0 C, autotdb probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **u500, Jan 21 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **115.19**

Relaxation Delay(s): **1**
Completed Scans **564**

DK-9-27

125.688 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 26.1 C -> actual temp = 27.0 C, autotx probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **mr400, Feb 27 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **4807.69**
Digital Res.(Hz/pt): **0.07**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **15.92**

Relaxation Delay(s): **0.1**
Completed Scans **16**

OB-1-115

399.978 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 25.9 C -> actual temp = 27.0 C, onenmr probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **i400, Feb 27 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **26954.2**
Digital Res.(Hz/pt): **0.21**

Acquisition Time(s): **1.002**
Hz per mm(Hz/mm): **93.43**

Relaxation Delay(s): **1**
Completed Scans **536**

OB-1-115

100.539 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 26.5 C -> actual temp = 27.0 C, autoxdb probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **mr400, Feb 27 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **4807.69**
Digital Res.(Hz/pt): **0.07**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **16.03**

Relaxation Delay(s): **0.1**
Completed Scans **16**

OB-1-107

399.978 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 25.9 C -> actual temp = 27.0 C, onenmr probe

Department of Chemistry, University of Alberta

Recorded on: **mr400, Feb 27 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **27173.9**
Digital Res.(Hz/pt): **0.21**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **92.94**

Relaxation Delay(s): **1**
Completed Scans **692**

OB-1-107
100.586 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 25.9 C -> actual temp = 27.0 C, onenmr probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **v700, Jan 30 2019**
Pulse Sequence: **PRESAT**

Sweep Width(Hz): **8389.26**
Digital Res.(Hz/pt): **0.13**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **28.58**

Relaxation Delay(s): **0.1**
Completed Scans **8**

DK-9-67

699.762 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **v700, Jan 30 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **36764.7**
Digital Res.(Hz/pt): **0.28**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **153.19**

Relaxation Delay(s): **1**
Completed Scans **512**

DK-9-67

175.971 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.5 C -> actual temp $\pm$ 27.0 C, coldid probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **v700, Apr 6 2019**
Pulse Sequence: **PRESAT**

Sweep Width(Hz): **8389.26**
Digital Res.(Hz/pt): **0.13**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **28.24**

Relaxation Delay(s): **0.1**
Completed Scans **8**

OB-1-193

699.762 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **v700, Apr 6 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **36764.7**
Digital Res.(Hz/pt): **0.28**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **153.19**

Relaxation Delay(s): **1**
Completed Scans **36**

OB-1-193

175.971 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **ibd5, Mar 16 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **6000.6**
Digital Res.(Hz/pt): **0.09**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **19.53**

Relaxation Delay(s): **0.1**
Completed Scans **64**

OB-1-151

498.118 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 26.9 C -> actual temp = 27.0 C, autotdb probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **ibd5, Mar 16 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **33826.6**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **0.998**
Hz per mm(Hz/mm): **113.27**

Relaxation Delay(s): **1**
Completed Scans: **1440**

OB-1-151

125.266 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 26.9 C -> actual temp = 27.0 C, autotx probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **u500, Apr 24 2019**
Pulse Sequence: **PRESAT**

Sweep Width(Hz): **6009.62**
Digital Res.(Hz/pt): **0.09**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **20.66**

Relaxation Delay(s): **0.1**
Completed Scans **8**

DK-10-123

499.787 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **u500, Apr 24 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **119.59**

Relaxation Delay(s): **1**
Completed Scans **604**

DK-10-123

125.685 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **u500, Feb 8 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **6009.62**
Digital Res.(Hz/pt): **0.09**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **20.13**

Relaxation Delay(s): **0.1**
Completed Scans **4**

OB-1-97

499.797 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 26.1 C -> actual temp = 27.0 C, autotx probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **u500, Feb 8 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **118.76**

Relaxation Delay(s): **1**
Completed Scans **392**

OB-1-97

125.688 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 26.1 C -> actual temp = 27.0 C, autotdb probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **v700, Nov 30 2018**
Pulse Sequence: **PRESAT**

Sweep Width(Hz): **8389.26**
Digital Res.(Hz/pt): **0.13**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **27.09**

Relaxation Delay(s): **0.1**
Completed Scans **8**

DK-8-109-B

699.762 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe

S-172

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **v700, Nov 30 2018**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **48076.9**
Digital Res.(Hz/pt): **0.37**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **152.03**

Relaxation Delay(s): **1**
Completed Scans **64**

DK-8-109-B

175.975 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **u500, Jan 30 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **6009.62**
Digital Res.(Hz/pt): **0.09**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **20.03**

Relaxation Delay(s): **0.1**
Completed Scans **8**

OB-1-81

499.797 MHz H1 1D in cdcl3 (ref. to CDCI3 @ 7.26 ppm) temp 26.1 C -> actual temp = 27.0 C, autoxdb probe

7d

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **u500, Jan 30 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **115**

Relaxation Delay(s): **1**
Completed Scans **264**

OB-1-81

125.688 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 26.1 C -> actual temp = 27.0 C, autotxdb probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **u500, Feb 13 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **6009.62**
Digital Res.(Hz/pt): **0.09**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **19.77**

Relaxation Delay(s): **0.1**
Completed Scans **16**

OB-1-101

499.797 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 26.1 C -> actual temp = 27.0 C, autotdb probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **u500, Feb 13 2019**
Pulse Sequence: **s2pul**Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**Acquisition Time(s): **1**
Hz per mm(Hz/mm): **114.26**Relaxation Delay(s): **1**
Completed Scans **216**

OB-1-101

125.688 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 26.1 C -> actual temp = 27.0 C, autotdb probe

S-177

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **ibd5, Apr 9 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **6000.6**
Digital Res.(Hz/pt): **0.09**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **19.63**

Relaxation Delay(s): **0.1**
Completed Scans **16**

OB-1-185

498.118 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 26.9 C -> actual temp = 27.0 C, autotx probe

S-178

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **ibd5, Apr 9 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **33826.6**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **0.998**
Hz per mm(Hz/mm): **113.85**

Relaxation Delay(s): **1**
Completed Scans: **720**

OB-1-185
125.266 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 26.9 C -> actual temp = 27.0 C, autotdb probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **u500, Feb 2 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **6009.62**
Digital Res.(Hz/pt): **0.09**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **19.75**

Relaxation Delay(s): **0.1**
Completed Scans **16**

OB-1-83

499.797 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 26.1 C -> actual temp = 27.0 C, autotdb probe

S-180

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **u500, Feb 2 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **116.8**

Relaxation Delay(s): **1**
Completed Scans: **3652**

OB-1-83

125.688 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 26.1 C -> actual temp = 27.0 C, autoxdb probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **u500, Feb 6 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **6009.62**
Digital Res.(Hz/pt): **0.09**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **20.45**

Relaxation Delay(s): **0.1**
Completed Scans **16**

OB-1-95

499.797 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 26.1 C -> actual temp = 27.0 C, autotx probe

S-182

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **u500, Feb 6 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **115.82**

Relaxation Delay(s): **1**
Completed Scans **552**

OB-01-95

125.688 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 26.1 C -> actual temp = 27.0 C, autotx probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **v700, Feb 5 2019**
Pulse Sequence: **PRESAT**

Sweep Width(Hz): **8389.26**
Digital Res.(Hz/pt): **0.13**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **27.44**

Relaxation Delay(s): **0.1**
Completed Scans **8**

DK-9-97

699.762 MHz H1 1D in cdcl3 (ref. to CDC13 @ 7.26 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **v700, Feb 2 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **36764.7**
Digital Res.(Hz/pt): **0.28**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **153.19**

Relaxation Delay(s): **1**
Completed Scans **368**

DK-9-97

175.971 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **u500, Apr 12 2019**
Pulse Sequence: **PRESAT**

Sweep Width(Hz): **6009.62**
Digital Res.(Hz/pt): **0.09**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **19.74**

Relaxation Delay(s): **0.1**
Completed Scans **8**

DK-10-103

499.787 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **u500, Apr 12 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **118.01**

Relaxation Delay(s): **1**
Completed Scans **52**

DK-10-103

125.685 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **v700, Apr 6 2019**
Pulse Sequence: **PRESAT**

Sweep Width(Hz): **8389.26**
Digital Res.(Hz/pt): **0.13**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **28.01**

Relaxation Delay(s): **0.1**
Completed Scans **8**

OB-01-195

699.762 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **v700, Apr 6 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **36764.7**
Digital Res.(Hz/pt): **0.28**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **153.19**

Relaxation Delay(s): **1**
Completed Scans: **20**

OB-01-195

175.971 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **ibd5, Mar 22 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **6000.6**
Digital Res.(Hz/pt): **0.09**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **19.85**

Relaxation Delay(s): **0.1**
Completed Scans **4**

OB-1-161

498.118 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 26.9 C -> actual temp = 27.0 C, autotx probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **ibd5, Mar 22 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **33826.6**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **0.998**
Hz per mm(Hz/mm): **113.49**

Relaxation Delay(s): **1**
Completed Scans **276**

OB-1-161

125.266 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 26.9 C -> actual temp = 27.0 C, autotdb probe

11

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **i600, Apr 26 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **7183.91**
Digital Res.(Hz/pt): **0.11**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **23.55**

Relaxation Delay(s): **0.1**
Completed Scans **16**

OB-2-19

599.926 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 26.2 C -> actual temp = 27.0 C, autoxid probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **ibd5, Apr 26 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **33826.6**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **0.998**
Hz per mm(Hz/mm): **112.88**

Relaxation Delay(s): **1**
Completed Scans **180**

OB-2-19

125.266 MHz C13{H1} 1D in d2o (ref. to external acetone @ 31.07 ppm) temp 26.9 C -> actual temp = 27.0 C, autotdb probe

12

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **v700, Apr 26 2019**
Pulse Sequence: **PRESAT**

Sweep Width(Hz): **6389.26**
Digital Res.(Hz/pt): **0.13**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **28.23**

Relaxation Delay(s): **0.1**
Completed Scans: **8**

DK-10-131

699.762 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **v700, Apr 26 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **36764.7**
Digital Res.(Hz/pt): **0.28**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **153.19**

Relaxation Delay(s): **1**
Completed Scans **1100**

DK-10-131

175.971 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **ibd5, Feb 18 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **6000.6**
Digital Res.(Hz/pt): **0.09**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **19.74**

Relaxation Delay(s): **0.1**
Completed Scans **16**

DK-9-131

498.118 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 26.9 C -> actual temp = 27.0 C, autotdb probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **u500, Apr 5 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **119.22**

Relaxation Delay(s): **1**
Completed Scans **96**

DK-9-131

125.685 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **u500, Feb 28 2019**
Pulse Sequence: **PRESAT**

Sweep Width(Hz): **6009.62**
Digital Res.(Hz/pt): **0.09**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **19.88**

Relaxation Delay(s): **0.1**
Completed Scans **8**

DK-9-155

499.787 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **u500, Feb 28 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **117.29**

Relaxation Delay(s): **1**
Completed Scans **512**

DK-9-155

125.685 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **u500, Mar 14 2019**
Pulse Sequence: **PRESAT**

Sweep Width(Hz): **6009.62**
Digital Res.(Hz/pt): **0.09**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **21.53**

Relaxation Delay(s): **0.1**
Completed Scans **8**

DK-9-187

499.787 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe

S-200

Recorded on: **u500, Mar 14 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **33783.8**
Digital Res.(Hz/pt): **0.26**

Acquisition Time(s): 1
Hz per mm(Hz/mm): 116.55

Relaxation Delay(s): 1
Completed Scans 72

125.685 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.7 C -> actual temp = 27.0 C, cold dual probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **v700, Mar 6 2019**
Pulse Sequence: **PRESAT**

Sweep Width(Hz): **8389.26**
Digital Res.(Hz/pt): **0.13**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **27.55**

Relaxation Delay(s): **0.1**
Completed Scans **8**

DK-9-177

699.762 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **v700, Mar 6 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **36764.7**
Digital Res.(Hz/pt): **0.28**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **153.19**

Relaxation Delay(s): **1**
Completed Scans **16**

DK-9-177

175.971 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **v700, Mar 9 2019**
Pulse Sequence: **PRESAT**

Sweep Width(Hz): **8389.26**
Digital Res.(Hz/pt): **0.13**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **27.73**

Relaxation Delay(s): **0.1**
Completed Scans **8**

DK-9-179

699.762 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **v700, Mar 9 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **36764.7**
Digital Res.(Hz/pt): **0.28**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **153.19**

Relaxation Delay(s): **1**
Completed Scans: **512**

DK-9-179

175.971 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **v700, Mar 14 2019**
Pulse Sequence: **PRESAT**

Sweep Width(Hz): **6389.26**
Digital Res.(Hz/pt): **0.13**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **26.71**

Relaxation Delay(s): **0.1**
Completed Scans **8**

DK-9-201

699.762 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe

Agilent Technologies

Department of Chemistry, University of Alberta

Recorded on: **v700, Mar 14 2019**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **36764.7**
Digital Res.(Hz/pt): **0.28**

Acquisition Time(s): **1**
Hz per mm(Hz/mm): **153.19**

Relaxation Delay(s): **1**
Completed Scans **5000**

DK-9-201

175.971 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) temp 27.5 C -> actual temp = 27.0 C, coldid probe

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