## **Supporting Information**

## Direct and Indirect Hyperpolarisation of Amines using paraHydrogen

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### 1 NMR polarisation transfer experiment data

#### 1.1 SABRE polarisation transfer method

The polarisation transfer experiments that are reported in this study were conducted in 5 mm NMR tubes that were equipped with a J. Young's tap. Samples for these polarisation transfer experiments were based on 5 mM solutions of [IrCl(COD)(IMes)] and the indicated substrate and loadings in methanol- $d_4$  or dichloromethane- $d_2$  (0.6 mL). The samples were degassed prior to the introduction of NH<sub>3</sub>. Subsequently, *para*hydrogen (*p*-H<sub>2</sub>) at a pressure of ca. 3 bar was added. Samples were then shaken for 10 s in the specified fringe field of an NMR spectrometer (9.4 T) before being rapidly transported into the magnet for subsequent interrogation by NMR spectroscopy. This whole process takes ca. 15 seconds to achieve.

## 1.2 SABRE-RELAY polarisation transfer method with NH<sub>3</sub>

The polarisation transfer experiments that are reported were conducted in similar 5 mm NMR tubes equipped with J. Young's valves. Samples were based on 5 mM solutions of [IrCl(COD)(IMes)], NH<sub>3</sub> (5-10 eq.), and the indicated additional substrate at the specified loading in dichloromethane- $d_2$  (0.6 mL). The samples were degassed prior to the introduction of p-H<sub>2</sub> at a pressure of ca. 3 bar. They were shaken for 10 s in the specified fringe field of an NMR spectrometer (9.4 T) before being rapidly transported into the magnet for subsequent interrogation by NMR spectroscopy.

#### **1.3 Polarisation factors**

For calculation of the <sup>1</sup>H and <sup>13</sup>C signal enhancements the following formula was used:

$$E = \frac{SI(pol)}{SI(unpol)}$$

Where, E = enhancement level, SI(pol) = signal of polarised sample, SI(unpol) = signal of unpolarised (reference) sample. Experimentally, both spectra were recorded on the same sample using identical acquisition parameters, including the receiver gain. The raw integrals of the relevant resonances in the polarised and unpolarised spectra were then used to determine the enhancement levels. The quoted values reflect the signal strength gain (fold) per proton/phosphorus nucleus in the specified group.

<sup>13</sup>C enhancements were calculated by taking the raw integral of the  ${}^{13}CD_2Cl_2$  peak observed from the solvent in the sample after equilibration inside the magnet for 1 minute.  $CD_2Cl_2$  was present in each sample at a concentration of 15.67 M and the resulting SABRE-Relay

hyperpolarised signal was then scaled according to the concentration of substrate in solution (given in section 2) to give the final enhancement value for  ${}^{13}C$ . These values were validated by further measurements on samples containing CHCl<sub>3</sub> as an internal standard.

## **1.4 NMR Spectrometer**

Spectra were typically acquired on a 400 MHz Bruker, Avance III console using a 5 mm BBI probe which was tuned to <sup>1</sup>H, <sup>13</sup>C, <sup>31</sup>P or <sup>15</sup>N as specified. Resonances are referenced relative to the residual proton signal of the indicated deuterated solvent.

## **1.5 Pulse Sequences**

## • Refocused INEPT

INEPT based measurements of <sup>13</sup>C and <sup>15</sup>N NMR spectra were recorded using a standard refocused INEPT experiment with decoupling during acquisition as outlined in Fig. S1. The evolution delay (d4) is optimised to  $1/2 \times J_{XH}$  and obtains in-phase X-magnetisation through the addition of a refocusing delay (d3) which is set to  $1/(6 \times J_{XH})$ . Values of  $J_{XH}$  are given in section 2.



Fig. S1: Ineptrd pulse sequence utilised for SABRE-RELAY derived heteronuclei detection.

## 2 Representative SABRE NMR spectra and enhancement factors

## • Ammonia

Fig. S2 reflects a series of single scan <sup>1</sup>H NMR spectra employing ammonia in  $CD_2Cl_2$ : a) <sup>1</sup>H NMR spectrum thermally polarised x16 vertical scale, b) <sup>1</sup>H SABRE NMR spectrum hyperpolarisation; using the conditions: [IrCl(COD)(IMes)] (5 mM),  $CD_2Cl_2$  (0.6 mL), NH<sub>3</sub>; <sup>1</sup>H enhancement (fold/per proton): 94-(NH<sub>3</sub>), x1 vertical scale.



Fig. S2a Thermally polarised <sup>1</sup>H NMR reference spectrum for ammonia in CD<sub>2</sub>Cl<sub>2</sub>





Fig. S2b <sup>1</sup>H SABRE enhanced NMR spectrum of ammonia in  $CD_2Cl_2$  (top) and expansion (bottom).

#### • Benzylamine

Fig. S3 reflects a series of single scan <sup>1</sup>H NMR spectra of benzylamine: a) <sup>1</sup>H NMR spectrum thermally polarised x8 vertical scale, b) <sup>1</sup>H SABRE NMR spectrum, c-f) <sup>1</sup>H-<sup>13</sup>C refocussed INEPT <sup>13</sup>C SABRE NMR spectra and d) <sup>1</sup>H NMR spectrum recorded after adding 1  $\mu$ l of D<sub>2</sub>O (max enhancement (per proton) of H<sub>2</sub>O 215 fold); conditions: [IrCl(COD)(IMes)] (5 mM), benzylamine (10 eq.), CD<sub>2</sub>Cl<sub>2</sub> (0.6 mL); Substrate concentration: 50 mM. <sup>1</sup>H NMR signal enhancement (fold/per proton): 72-(NH<sub>2</sub>), 56-(CH<sub>2</sub>) and 194-(Ph).



Fig. S3a Thermally polarised <sup>1</sup>H NMR reference spectrum of benzylamine



Fig. S3b <sup>1</sup>H SABRE enhanced NMR spectrum of benzylamine (top) and expansion (bottom)



Fig. S3c <sup>1</sup>H-<sup>13</sup>C refocussed INEPT <sup>13</sup>C SABRE enhanced NMR spectrum of benzylamine  $(J_{XH} = 10 \text{ Hz}, \text{B} = 60 \text{ G})$ 



Fig. S3d <sup>1</sup>H-<sup>13</sup>C refocussed INEPT <sup>13</sup>C SABRE enhanced NMR spectrum of benzylamine  $(J_{XH} = 10 \text{ Hz}, \text{B} = \mu\text{-magnetic shield})$ 



Fig. S3e <sup>1</sup>H-<sup>13</sup>C refocussed INEPT <sup>13</sup>C SABRE enhanced NMR spectrum of benzylamine  $(J_{XH} = 135 \text{ Hz}, \text{B} = 60 \text{ G})$ 



Fig. S3f <sup>1</sup>H-<sup>13</sup>C refocussed INEPT <sup>13</sup>C SABRE enhanced NMR spectrum of benzylamine  $(J_{XH} = 135 \text{ Hz}, \text{B} = \mu\text{-magnetic shield})$ 



Fig. S3g <sup>1</sup>H SABRE enhanced NMR spectrum for benzylamine after adding 1  $\mu l$  of  $D_2O$ 

## • Benzylamine-<sup>15</sup>N

Fig. S4 reflects a series of single scan NMR spectra of benzylamine- $^{15}$ N: a) <sup>1</sup>H NMR spectrum thermally polarised x16, b) <sup>1</sup>H SABRE NMR spectrum and c) <sup>15</sup>N SABRE NMR spectrum using conditions: [IrCl(COD)(IMes)] (5 mM), benzylamine- $^{15}$ N (10 eq.), CD<sub>2</sub>Cl<sub>2</sub> (0.6 mL); Substrate concentration: 50 mM. <sup>1</sup>H enhancement (fold/per proton): 33-(NH<sub>2</sub>), 34-(CH<sub>2</sub>) and 52-(Ph)



Fig. S4a Thermally polarised <sup>1</sup>H NMR reference spectrum of benzylamine-<sup>15</sup>N



Fig. S4b <sup>1</sup>H SABRE enhanced NMR spectrum of benzylamine-<sup>15</sup>N (top) and expansion (bottom)



Fig. S4c <sup>15</sup>N SABRE enhanced NMR spectrum of benzylamine-<sup>15</sup>N (B = 0 G)

#### • Phenylethylamine

Fig. S5 reflects a series of single scan NMR spectra of phenylethylamine: a) <sup>1</sup>H NMR spectrum thermally polarised x8, b) <sup>1</sup>H SABRE NMR spectrum and c) INEPT <sup>13</sup>C SABRE NMR spectrum using conditions: [IrCl(COD)(IMes)] (5 mM), phenylethylamine (10 eq.),  $CD_2Cl_2$  (0.6 mL); Substrate concentration: 50 mM. <sup>1</sup>H enhancement (fold/per proton): 108-(NH<sub>2</sub>), 45- (CH<sub>2</sub>), 50-(NCH<sub>2</sub>) and 92-(Ph)



Fig. S5a Thermally polarised <sup>1</sup>H NMR reference spectrum for phenylethylamine





Fig. S5c INEPT <sup>13</sup>C SABRE enhanced NMR spectrum of phenylethylamine ( $J_{XH}$  = 122 Hz, B = 60 G)

#### • Phenoxyethylamine

Fig. S6 reflects a series of single scan NMR spectra of phenoxyethylamine: a) <sup>1</sup>H NMR spectrum thermally polarised x16, b) <sup>1</sup>H SABRE NMR spectrum and c) INEPT <sup>13</sup>C SABRE NMR spectrum using conditions: [IrCl(COD)(IMes)] (5 mM), phenoxyethylamine (10 eq.),  $CD_2Cl_2$  (0.6 mL); Substrate concentration: 50 mM. <sup>1</sup>H enhancement (fold/per proton): 99-(NH<sub>2</sub>), 47- (NCH<sub>2</sub>), 147-(CH<sub>2</sub>O) and 8-(Ph)



Fig. S6a Thermally polarised <sup>1</sup>H NMR reference spectrum of phenoxyethylamine







Fig. S6c INEPT <sup>13</sup>C SABRE enhanced NMR spectrum of phenoxyethylamine ( $J_{XH}$  = 165 Hz, B = 60 G)

#### • Isobutylamine

Fig. S7 reflects a series of single scan NMR spectra of isobutylamine: a) <sup>1</sup>H NMR spectrum thermally polarised x8 and b) <sup>1</sup>H SABRE NMR spectrum using conditions: [IrCl(COD)(IMes)] (5 mM), isobutylamine (10 eq.),  $CD_2Cl_2$  (0.6 mL); Substrate



concentration: 50 mM. <sup>1</sup>H enhancement (fold/per proton): 187- (NH<sub>2</sub>), 83-(-CH<sub>2</sub>), 65-(CH<sub>-</sub>) and 124-(-CH<sub>3</sub>)

Fig. S7a Thermally polarised <sup>1</sup>H NMR reference spectrum for isobutylamine





Fig. S7b<sup>1</sup>H SABRE enhanced NMR spectrum of isobutylamine (top) and expansion (bottom)

#### • Allylamine

Fig. S8 reflects a series of single scan NMR spectra of allylamine: a) <sup>1</sup>H NMR spectrum thermally polarised x4 and b) <sup>1</sup>H SABRE NMR spectrum using conditions: [IrCl(COD)(IMes)] (5 mM), allylamine (10 eq.),  $CD_2Cl_2$  (0.6 mL); Substrate concentration: 50 mM. <sup>1</sup>H enhancement (fold/per proton): 76-(NH<sub>2</sub>), 131-(-CH<sub>2</sub>N), 178-(CH<sub>-</sub>) and 158-(-CH<sub>2</sub>)



Fig. S8a Thermally polarised <sup>1</sup>H NMR reference spectrum of allylamine





Fig. S8b <sup>1</sup>H SABRE enhanced NMR spectrum of allylamine (top) and expansion (bottom)

#### • Tryptamine

Fig. S9 reflects a series of single scan NMR spectra of tryptamine: a) <sup>1</sup>H NMR spectrum thermally polarised x8 and b) <sup>1</sup>H SABRE NMR spectrum using conditions: [IrCl(COD)(IMes)] (5 mM), tryptamine (10 eq.),  $CD_2Cl_2$  (0.6 mL); Substrate concentration: 50 mM. <sup>1</sup>H enhancement (fold/per proton): 21-(-NH<sub>2</sub>), 21-(CH<sub>2</sub>-N), 8-(-CH<sub>2</sub>-), 14-(-CH-), 7-(-NH-) and 28-(Ph)



Fig. S9a Thermally polarised <sup>1</sup>H NMR reference spectrum of tryptamine





Fig. S9b<sup>1</sup>H SABRE enhanced NMR spectrum of tryptamine (top) and expansion (bottom)

#### **3** Representative SABRE NMR spectra and enhancement factors using a co-ligand

#### • Aniline

Fig. S10 reflects a series of single scan NMR spectra of aniline: a) <sup>1</sup>H NMR spectrum thermally polarised x8, b) <sup>1</sup>H SABRE NMR spectrum c) <sup>13</sup>C SABRE NMR spectrum and d) INEPT <sup>13</sup>C SABRE NMR spectrum using conditions: [IrCl(COD)(IMes)] (5 mM), aniline (10 eq.), CD<sub>2</sub>Cl<sub>2</sub> (0.6 mL); Substrate concentration: 50 mM and MeCN as co-ligand (8 fold excess). <sup>1</sup>H enhancement (fold/per proton): 306-(NH<sub>2</sub>) and 193-(Ph)



Fig. S10a Thermally polarised <sup>1</sup>H NMR reference spectrum of aniline





Fig. S10b <sup>1</sup>H SABRE enhanced NMR spectrum of aniline (top) and expansion (bottom)



Fig. S10c<sup>13</sup>C SABRE enhanced NMR spectrum of spectrum for aniline



Fig. S10d INEPT <sup>13</sup>C SABRE enhanced NMR spectrum of phenoxyethylamine ( $J_{XH}$  = 120 Hz, B = 60 G)

#### 4 Representative SABRE-RELAY NMR spectra and enhancement factors

#### • Isopropylamine

Fig. S11 reflects a series of single scan NMR spectra of isopropylamine: a) <sup>1</sup>H NMR spectrum thermally polarised x16, b) <sup>1</sup>H SABRE NMR spectrum and c) <sup>13</sup>C SABRE NMR spectrum using conditions: [IrCl(COD)(IMes)] (5 mM), isopropylamine (10 eq.), CD<sub>2</sub>Cl<sub>2</sub> (0.6 mL); Substrate concentration: 50 mM. <sup>1</sup>H enhancement (fold/per proton): 220- (NH<sub>2</sub>), 27- (CH) and 150-(CH<sub>3</sub>)



Fig. S11a Thermally polarised <sup>1</sup>H NMR reference spectrum for isopropylamine



Fig. S11b<sup>1</sup>H SABRE enhanced NMR spectrum of isopropylamine (top) and expansion (bottom)



Fig. S11c INEPT <sup>13</sup>C SABRE enhanced NMR spectrum of isopropylamine ( $J_{XH}$  = 122 Hz, B = 60 G)

## • Dibenzylamine

Fig. S12 reflects a series of single scan NMR spectra of dibenzylamineamine: a) <sup>1</sup>H NMR spectrum thermally polarised x64, b) <sup>1</sup>H SABRE NMR spectrum c) <sup>13</sup>C SABRE NMR spectrum and d) INEPT <sup>13</sup>C SABRE NMR spectrum using conditions: [IrCl(COD)(IMes)] (5 mM), dibenzylamine (10 eq.), CD<sub>2</sub>Cl<sub>2</sub> (0.6 mL); Substrate concentration: 50 mM. <sup>1</sup>H enhancement (fold/per proton): 274-(NH-), 200-(CH<sub>2</sub>) and 275-(Ph).



Fig. S12a Thermally polarised <sup>1</sup>H NMR reference spectrum of dibenzylamineamine



Fig. S12b <sup>1</sup>H SABRE enhanced NMR spectrum of dibenzylamineamine (top) and expansion (bottom)



Fig. S12c <sup>13</sup>C SABRE enhanced NMR spectrum of dibenzylamineamine



Fig. S12d INEPT <sup>13</sup>C SABRE enhanced NMR spectrum of dibenzylamineamine ( $J_{XH}$  = 122 Hz, B = 60 G)

#### • Aniline

Fig. S13 reflects a series of single scan NMR spectra of aniline: a) <sup>1</sup>H NMR spectrum thermally polarised x8 and b) <sup>1</sup>H SABRE NMR spectrum using conditions: [IrCl(COD)(IMes)] (5 mM), aniline (10 eq.),  $CD_2Cl_2$  (0.6 mL); Substrate concentration: 50 mM. <sup>1</sup>H enhancement (fold/per proton): 150-(NH<sub>2</sub>) and 9-(Ph)



Fig. S13a Thermally polarised <sup>1</sup>H NMR reference spectrum of aniline

![](_page_29_Figure_2.jpeg)

![](_page_30_Figure_0.jpeg)

![](_page_30_Figure_1.jpeg)

## 5 Optimisation of the SABRE polarisation level of BnNH<sub>2</sub>

## 5.1 Effect of varying the pressure of *p*-H<sub>2</sub>

Increasing the pressure of p-H<sub>2</sub> was shown to improve the observed signal gains for the protons of BnNH<sub>2</sub> as shown in Figure S14.

![](_page_31_Figure_0.jpeg)

Fig. S14: Effect of p-H<sub>2</sub> pressure on the SABRE polarisation of BnNH<sub>2</sub> (10 eq.) using **1** (5 mM) in dichloromethane- $d_2$  solution

#### 5.2 Effect of Temperature of SABRE catalysis

Increasing temperature at which the SABRE transfer occurs increased the observed signal gains of BnNH<sub>2</sub> as shown in Figure S15.

![](_page_31_Figure_4.jpeg)

Fig. S15: Effect of temperature on the SABRE polarisation of BnNH<sub>2</sub> with 1 in dichloromethane- $d_2$  under 3-bar p-H<sub>2</sub> and transfer at 60 G.

#### 6 Ligand exchange dynamics of 2-NH<sub>3</sub> and 2-BnNH<sub>2</sub>

The kinetic behaviour of  $2-NH_3$  and  $2-BnNH_2$  under 3 bar  $H_2$  was examined using well established exchange spectroscopy methods as follows:

A series of exchange spectroscopy (EXSY) measurements were performed to probe the dynamic behaviour of these systems. This process involved the selective excitation of a single resonance and the subsequent measurement of a <sup>1</sup>H NMR spectrum at time, t, after the initial pulse. The resulting measurements consisted of a series of data arrays such that t is varied

typically between 0.1 to 1.0 s, to encode the reaction profile. Integrals for the interchanging peaks in the associated <sup>1</sup>H EXSY spectra were obtained and converted into a percentage of the total detected signal. These data were then analysed as a function of the mixing time according to a differential kinetic model.

Ligand	Temperature / K	Rate of Dissociation / s <sup>-1</sup>
Equatorial NH <sub>3</sub>	298	$0.32 \pm 0.01$
Hydride	298	$1.65 \pm 0.02$
Equatorial NH <sub>3</sub>	308	$10.42 \pm 0.06$
Hydride	308	$1.41 \pm 0.01$

Table S1: Observed rate of ligand loss from 2-NH<sub>3</sub> at varying temperatures.

Ligand	Temperature / K	Rate of Dissociation / s <sup>-1</sup>
Equatorial NH <sub>3</sub>	298	$3.33 \pm 0.03$
Hydride	298	$2.84 \pm 0.05$
Equatorial NH <sub>3</sub>	308	$9.85 \pm 0.04$

Table S2: Observed rate of ligand loss from 2-BnNH<sub>2</sub> at the indicated temperature.

## 7 NMR Characterisation data for the active SABRE catalysts featuring in this paper

## 7.1 NMR characterisation data for 2-NH<sub>3</sub>

![](_page_33_Figure_2.jpeg)

<b>Resonance number</b>	<sup>1</sup> H (ppm)	<sup>13</sup> C (ppm)	<sup>15</sup> N (ppm)
1		153.8	
2			190.83
3	6.80	121.5	
4		138.11	
5		135.25	
6	7.00	129.00	
7		138.64	
8	2.10	18.30	
9	2.35	20.78	
10	-23.61		
	2.21 (br, s, $J_{\rm NH} = 67$		
11	Hz)		-34.9
12	$2.84$ (br, s, $J_{\rm NH} = 69$		-46.5
12	Hz)		10.0

## 7.2 NMR characterisation data for 2-BnNH<sub>2</sub>

![](_page_34_Figure_1.jpeg)

Resonance	<sup>1</sup> H (ppm)	<sup>13</sup> C (ppm)	<sup>15</sup> N (ppm)
number			
1		153.8	
2			192.83
3	6.80	121.73	
4		138.11	
5		135.25	
6	6.90	129.29	
7		138.64	
8	2.17	18.30	
9	2.21	20.78	
10	-23.95		
	5.00 (br, dt, $J_{HH}$ = 5 and 11 Hz, 2H, $J_{15NH}$ =		
11	68 Hz)		-6.4
	2.30 (br, t, $J_{HH}$ = 11 Hz, 2H, $J_{15NH}$ = 68 Hz)		
12	$3.65 (dt, J_{HH} = 5 and 15 Hz, 2 H), 3.30 (ddd,$	52.14	
12	$J_{HH} = 3$ , 12 and 15 Hz, 2H)	55.14	
13		141.8	
14	7.35 (d, $J_{HH} = 5 Hz$ )	128.33	
15	7.25 (t, $J_{HH} = 5 Hz$ )	126.88	
16	~7.28 - overlap	-	
17	$4.25 (t, J_{HH} = 7 Hz, 2H, J_{15NH} = 69 Hz)$		-13.4
18	$3.85 (t, J_{HH} = 7 Hz, 2H)$	57.26	
19		-	
20	7.30	128.35	
21	~7.28 - overlap	-	
22	~7.28 - overlap	-	

## 7.3 NMR characterisation data for 2-PEA

![](_page_35_Figure_1.jpeg)

Resonance number	<sup>1</sup> H (ppm)	<sup>13</sup> C (ppm)	<sup>15</sup> N (ppm)
1		153.8	
2			192.21
3	6.75	121.73	
4		138.14	
5		135.06	
6	7.0	129.4	
7		138.64	
8	2.17	18.30	
9	2.21	21.10	
10	-23.95		
11	4.42.00 (t, $J_{HH} = 11$ Hz, 2H, $J_{15NH} = 68$ Hz) 2.11 (br, t, $J_{HH} = 11$ Hz, 2H, $J_{15NH} = 68$ Hz)		-7.2
12	2.41 (dt, J <sub>HH</sub> = 9 and 11 Hz, 2 H), 3.30 (2H, overlap)	50.7	
13			
14		141.8	
15	$7.32 (dd, J_{HH} = 7 Hz)$	128.33	
16	7.25 (t, $J_{\rm HH}$ = 7 Hz)	126.88	
17	~7.28 - overlap	-	
18	$3.685 (t, J_{HH} = 5.9 \text{ Hz}, 2\text{H}, J_{15\text{NH}} = 69 \text{ Hz})$		-16.2
19	2.78 (t, J <sub>HH</sub> = 6.15 Hz, 2H)	53.9	
20		-	
21	7.32	128.33	
22	~7.2 - overlap	-	
23	~7.12 - overlap	-	

# 7.4 NMR characterisation data for [Ir(H)<sub>2</sub>(IMes)(aniline)<sub>2</sub>(MeCN)]Cl

![](_page_36_Figure_1.jpeg)

<b>Resonance number</b>	<sup>1</sup> H (ppm)	<sup>13</sup> C (ppm)	<sup>15</sup> N (ppm)
1		153.8	
2			193.75
3	6.90	121.5	
4		138.11	
5		135.25	
6	7.00	129.00	
7		138.64	
8	2.10	18.30	
9	2.35	20.78	
10	-24.72	-	-
	$4.34 (d, J_{\rm HH} = 8 \text{ Hz}),$	-	-25
11	$8.00 (d, J_{\rm HH} = 8  {\rm Hz})$		
12			
13	$6.50 (d, J_{\rm HH} = 7  {\rm Hz})$		
14	8.27 (t, $J_{HH}$ = 7 Hz)		
15	$6.78 (d, J_{\rm HH} = 7  {\rm Hz})$		
16			176
	0.95 (s)		
17			