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1,8-Bis(dimethylamino)-2-(4-methoxyphenyl)naphthalene: An electrospray-active analogue for η^6-coordinating ligands

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ABSTRACT

1,8-Bis(dimethylamino)-2-(4-methoxyphenyl)naphthalene (**1**) was synthesized for use as an η^6-coordinating ligand with a chargeable tag for ESI-MS analysis. The pK_a of the proton sponge moiety was essentially unaffected by addition of the anisole group (pK_a = 18.2 ± 0.7 at 297 K in CD_3CN). Cr(η^6-**1**)$(CO)_3$ (**2**) was prepared to demonstrate the utility of (**1**) as an ESI tag and **2** was easily visible by ESI(+)-MS. A single crystal structure of the complex showed η^6-binding of the ligand as expected with the charged moiety being far removed from the metal centre. The gas-phase reactivity of **2** was found to match that of the analogous uncharged chromium carbonyl complex $Cr(C_6H_5OCH_3)(CO)_3$ indicating that **1** has potential as a chargeable tag for the study of organometallic complexes and their reactions by ESI-MS.

1. Introduction

While electrospray ionization mass spectrometry (ESI-MS) is perhaps most widely known for its ability to manipulate and analyze large biomolecules, the technique is also steadily gaining recognition as a valuable tool for the study of organometallic complexes and their reactivity. The first experiments in this area were by Berman in 1991 in which ESI-MS was used to study environmentally relevant organoarsenic ions [1]. Since then, ESI-MS has been used to identify and monitor organometallic ions that play key roles in a variety of catalytic transformations including oxidation [2–5], hydrogenation [6–8], hydrosilylation [9,10] and carbon–carbon bond forming reactions [11–20]. ESI is especially well-suited to the study of organometallic catalysis because it allows charged species to be observed (1) at very low concentrations, (2) in complex mixtures and (3) in polar or non-polar solvents [21]. In addition, electrospray ionization (ESI) is a “soft ionization” technique meaning that little or no fragmentation of fragile organometallic ions is observed, and only species that are already charged in solution (or contain an easily charged site) can be detected. While this last point allows for very low detection limits (most common solvents are neutral and therefore invisible to ESI-MS), it also causes a problem when the organometallic species of interest is not charged. In some cases it is possible to study these formally neutral complexes by exploiting facile ionization pathways that are commonly available to organometallic complexes; for example, protonation of a basic site, loss of a halide, or oxidation of a low valent metal. However, many interesting species do not have an easily accessible ionization pathway, or when they do spectra may be severely complicated by multiple competing pathways.

To address this problem, charged “tags” have been employed to make otherwise neutral organometallic complexes amenable to ESI-MS. Quaternary ammonium [22–25], pyridinium [26], phosphonium [27,28] or sulfonate [7,29,30] bearing ligands are common, and tags are often derived from ligands designed for water solubility.

Another option is to use chargeable tags. Chargeable tags are neutral molecules that contain a functional group of interest, and at a secondary site bind either and acid or a base to become charged. In addition to the benefits conferred by charged tags, chargeable tags (1) minimize any effect the charged group may have on reactivity (since the charged tag is in equilibrium with its neutral counterpart), and (2) can be designed so that their solubility is pH dependant, thus making them potentially useful for catalyst recovery. The first to use such a tag was Canary in 1994 when he investigated the intermediates involved in the Suzuki reaction by

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Abbreviation: PS, Proton Sponge®; EDESI, energy-dependent electrospray ionization.

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employing pyridyl bromide as a substrate [31]. The substrate was easily charged through protonation of the ring nitrogen and its complexes were readily observable by ESI-MS.

We have developed a number of chargeable ligands derived from the strong base 1,8-bis(dimethylamino)naphthalene also known as “Proton Sponge®” (PS) that serve as analogues for commonly used organometallic ligands and are especially suited to act as chargeable tags [32,33]. First studied by Alder and coworkers, Proton Sponge® is a very strong, non-nucleophilic base (pK_a = 12.34 in H_2O) which acts to selectively bind H^+ by forming intramolecular hydrogen bonds with both nitrogen centres in the molecule to give the charged $[N{\cdots}H{\cdots}N]^+$ moiety. The high basicity of Proton Sponge® is thermodynamic in nature and arises from steric clashing of the methyl groups on the two nitrogen atoms which causes the nitrogen lone pairs to point toward each other. The lone pair–lone pair repulsion in turn leads to a twisted naphthalene backbone. Protonation of the neutral molecule returns the system to a more optimal geometry and forms strong intramolecular hydrogen bonds between the proton and both nitrogen lone pairs.

Steric crowding of the basic site by the methyl groups is severe and gives Proton Sponge® its non-nucleophilic tendencies: only proton can fit into the small pocket between nitrogen atoms. The overall effect is that Proton Sponge® is approximately six orders of magnitude more basic than typical aromatic amines, and can selectively trap adventitious protons to become charged.

Arenes are used extensively as η^6-ligands in organometallic chemistry. Their ability to change hapticity and expose an active metal centre makes them excellent ligands for catalyst precursors [34]. They have been used in hydrogenation and hydration reactions, olefin metathesis, cycloaddition and polymerization reactions [34,35]. For this reason the electrospray-active analogue of methoxybenzene was selected as a target compound. Herein we report the synthesis of the novel ligand 1,8-bis(dimethylamino)-2-(4-methoxyphenyl)naphthalene (**1**) and demonstrate its usefulness as an electrospray tag by investigating the unimolecular gas-phase reactivity of its chromium carbonyl complex ($Cr(CO)_3$(**1**)) (**2**).

2. Materials and methods

2.1. General information

Dry solvents were obtained from a solvent purification system. All solvents were HPLC grade. Reagents were purchased from Aldrich and used without further purification except for Proton Sponge® which was recrystallized from hot methanol before use. Reactions performed under nitrogen were carried out using standard Schlenk techniques. All electrospray mass spectra were collected using a Micromass Q-TOF *micro* instrument. Capillary voltage was set to 2900 V, source and desolvation temperatures were at 80 °C and 150 °C, respectively. Samples were infused via syringe pump at 10 μL min^{-1}. NMR spectra were recorded on a Bruker AC-300 spectrometer. Chemical shifts are quoted in ppm using internal references of $CHCl_3$ (^1H δ 7.26 ppm) or CH_3CN (^1H δ 1.94 ppm) where appropriate. Melting points were recorded on a Gallenkamp Melting Point Apparatus and are uncorrected. IR spectra were recorded using a solution cell in a Perkin Elmer Spectrum 1000 FT-IR spectrometer. X-Ray crystallographic data was collected by Dr. Robert MacDonald (University of Alberta) and Dr. Allen Oliver (University of Notre Dame). See Supplementary information for details.

2.2. Preparation of 1,8-bis(dimethylamino)-2-(4-methoxyphenyl) naphthalene (**1**)

{1,8-Bis(dimethylamino)naphthalene-2-yl}bromide was synthesized according to related literature [33]. Under a nitrogen atmosphere, {1,8-bis(dimethylamino)naphthalene-2-yl}bromide (5.00 g, 0.017 mol) and 4-methoxyphenylboronic acid (2.74 g, 0.018 mol) were dissolved in *n*-propanol (30 mL) with stirring. Distilled water (6 mL), sodium bicarbonate (10 mL, 2 M), palladium(II) acetate (0.034 g, 0.051 mmol), and triphenylphosphine (0.21 g, 0.8 mmol) were added consecutively and the mixture was refluxed overnight. The reaction mixture was cooled to room temperature and quenched by the dropwise addition of deionized water (21 mL) with stirring in air. The mixture was extracted twice with ethylacetate and the organic layer was washed with sodium bicarbonate (5% w/v, 2×) and brine (2×), and dried with sodium sulfate and activated charcoal (3 g). The mixture was filtered through a 3 cm bed of celite and the filtrate was reduced by rotary evaporation. Hexanes were added to yield the product as a white ppt in 41% yield (2.27 g). ^1H NMR (300 MHz, $CDCl_3$) δ_H **1**: 7.50–6.97 (m, aromatic protons, 9H), 3.88 (s, $OC\underline{H}_3$, 3H), 2.78 (s, $N(C\underline{H}_3)_2$, 6H), 2.63 (s, $N(C\underline{H}_3)_2$, 6H). ^{13}C NMR (400 MHz, CDCl3) δ_C **1**: 158.3, 137.3, 130.5, 130.2, 125.5, 123.1, 122.5, 113.7 (aromatic carbons); 55.5 ($O\underline{C}H_3$); 45.4 ($N(\underline{C}H_3)_2$); 45.1 ($N(\underline{C}H_3)_2$). ESI-MS (MeOH) *m*/*z*: 321.1983 ([**1** + H]$^+$, model $C_{21}H_{24}ON_2$ 321.1967). mp: 105–107 °C.

2.3. Preparation of 1,8-bis(dimethylamino)-2-(4-methoxyphenyl) naphthalene hydrofluoroborate ([**1**H]$^+$[BF_4]$^-$)

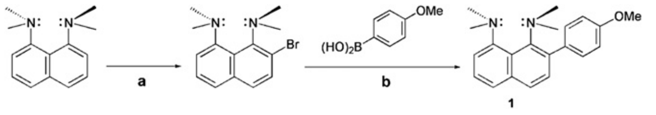
1 (0.0020 g, 4.9 μmol) was dissolved in ethanol and tetrafluoroboronic acid (0.004 mL, 48% in water) was added dropwise. Slow evaporation of solvent yielded colourless crystals (98%, 0.0039 g). ^1H NMR (300 MHz, $CDCl_3$) δ_H [**1**H]$^+$: 19.4 (s, H^+, 1H), 8.00–6.97 (m, aromatic protons, 9H), 3.79 (s, $OC\underline{H}_3$, 3H), 3.04 (s, $N(C\underline{H}_3)_2$, 6H), 2.79 (s, $N(C\underline{H}_3)_2$, 6H).

2.4. pK_a determination of **1**: ^1H transprotonation experiments [36]

Equimolar amounts of $[PS{\cdot}H]^+[BF_4]^-$ (0.0040 g, 0.013 mmol) and **1** (0.0042 g, 0.013 mmol) were dissolved together in dry CD_3CN (0.7 mL), allowed to stand overnight, and a ^1H NMR spectrum was recorded. The experiment was repeated four separate times. The average integrations for the coordinated proton of the two protonated species were (CD_3CN, 300 MHz) δ_H: 19.49 (**1**$\cdot H^+$, $\int$1.00), 18.67 ($PS{\cdot}H^+$, $\int$1.01) (Std. Dev. = 0.04, % RSD = 4). The first pK_a of **1** (CD_3CN) was determined by calculation using the ratio of the integrations of the two peaks and the known pK_a of PS (18.18 in CH_3CN) [37] Analysis of the equilibrium gave $K_{a1} = K_{aPS}/K_{eq}$ where K_{aPS} (known) = $[PS][H^+]/[PS{\cdot}H^+] = 1.514 \times 10^{18}$, $K_{a1} = [\mathbf{1}][H^+]/[\mathbf{1}{\cdot}H^+]$ and K_{eq} (exptl.) = $[\mathbf{1}{\cdot}H^+][PS]/[\mathbf{1}][PS{\cdot}H^+] = (\int \mathbf{1}{\cdot}H/\int PS{\cdot}H)^2 = 0.98$ [38]. $K_{a1} = 1.514 \times 10^{18}/0.98 = (1.54 \pm 0.06) \times 10^{18}$. p$K_a$ of **1** = 18.2 ± 0.7 in CD_3CN.

2.5. Preparation of chromium(**1**) tricarbonyl (**2**)

Under a nitrogen atmosphere, dry THF (0.13 mL) and dibutylether (2.5 mL) were added to **1** (0.1005 g, 0.314 mmol) and chromium hexacarbonyl (0.0521 g, 0.233 mmol). The mixture was refluxed for 24 h. The air-sensitive solution was quickly filtered in air and the solvent was removed by rotary evaporation to yield a yellow oil (28%, 0.030 g). Product was recrystallized under inert



Scheme 1. Synthesis of **1**. a) NBS/THF/−78 °C. b) $Pd(OAc)_2/PPh_3/NaCO_3H$/*n*-propanol/water. Reflux 12 h.

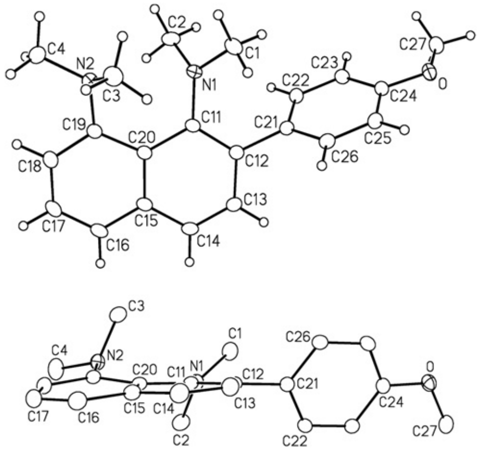


Fig. 1. Single crystal X-ray structure of 1,8-bis(dimethylamino)-2-(4-methoxyphenyl) naphthalene (**1**). Side- (top) and edge-on (bottom) perspectives of the naphthalene rings.

atmosphere from a solution of toluene by evaporation. ^{1}H NMR (300 MHz, $CDCl_3$) δ_H **2**: 7.47–7.05 (m, aromatic protons, 5H), 5.74 (d, anisole aromatic protons, 2H), 5.20 (d, anisole aromatic protons, 2H), 3.78 (s, $OC\underline{H}_3$, 3H), 2.74 (s, $N(C\underline{H}_3)_2$, 6H), 2.73 (s, $N(C\underline{H}_3)_2$, 6H). ν_{CO} bands: 1964 cm^{-1}, 1890 cm^{-1}. ESI-MS (MeOH) m/z: 457.1224 ([**2** + H]$^+$, model $C_{24}H_{24}O_4N_2Cr$ 457.1220). mp: 167–169 °C.

3. Results and discussion

3.1. Synthesis and characterization of *1*

1 was synthesized via bromination of Proton Sponge® at the two position as previously described by Farrer [33] to give {1,8-bis(dimethylamino)naphthalene-2-yl} bromide which was then coupled with 4-methoxyphenylboronic acid via a Suzuki cross-coupling reaction (Scheme 1).

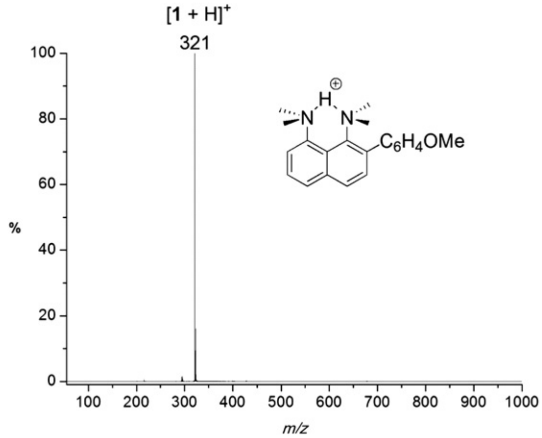


Fig. 3. ESI(+)-MS of **1** ([**1** + H]$^+$ = m/z 321) in dichloromethane spiked with formic acid.

After aqueous workup, the white crystalline product was isolated in 41% yield, and a single crystal structure of **1** was obtained (Fig. 1). In its unprotonated form the Proton Sponge® moiety deviates from planarity as expected due to mutual repulsion of the lone pairs on each nitrogen atom. This can be seen in the torsion angles of −8.78° for N(1)–C(11)–C(20)–C(19) and −9.63° for N(2)–C(19)–C(20)–C(11). The anisole group lies slightly off perpendicular to the plane of the naphthalene ring (torsion angle: C(13)–C(12)–C(21)–C(22) = 107.50°). We expected metal coordination to occur on the side of the arene ring that is furthest from the methyl groups on Proton Sponge®.

The approximate pK_a of **1** was determined by NMR trans-protonation experiments [39]. A deuterated acetonitrile solution

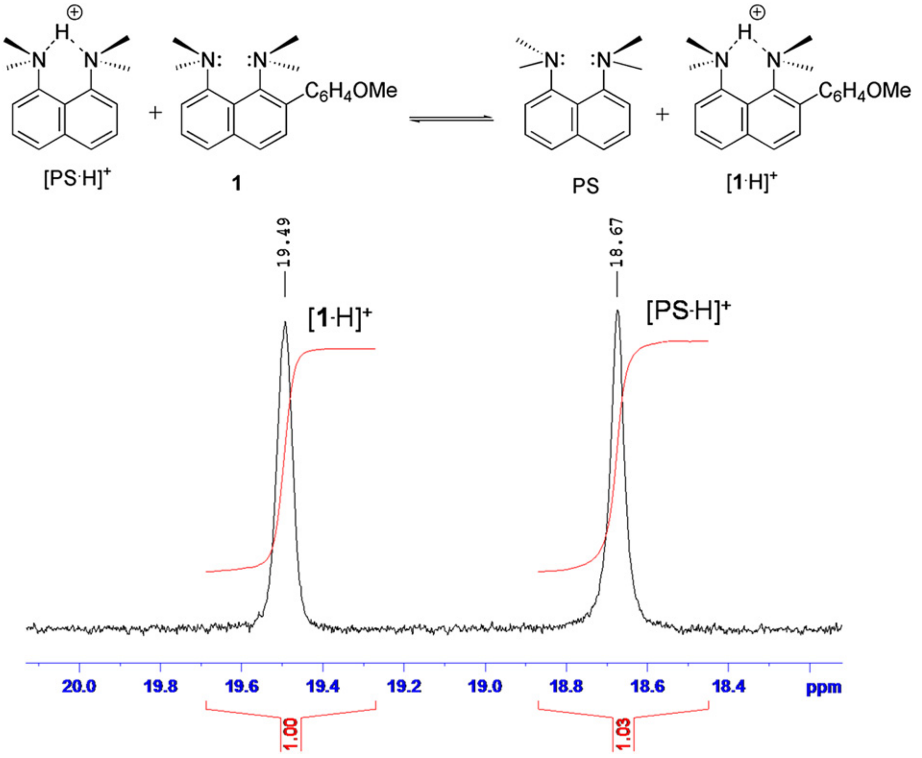


Fig. 2. : ^{1}H NMR spectrum (18–20 ppm) of an equimolar mixture of [PS·H]$^+$ and **1** in deuterated acetonitrile.

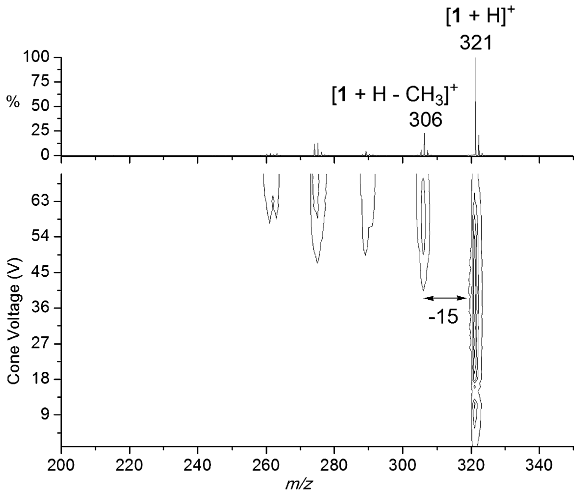


Fig. 4. EDESI of **1**.

containing equimolar amounts of unprotonated **1** and protonated Proton Sponge® ($[PS\cdot H]^+[BF_4]^-$) was allowed to come to equilibrium overnight and a ^1H NMR experiment was performed. The solubility of **1**, PS, $[\mathbf{1}\cdot H]^+[BF_4]^-$ and $[PS\cdot H]^+[BF_4]^-$ in acetonitrile were all carefully tested beforehand since any insolubility would lead to inaccurate signal intensities and therefore an inaccurate pK_a value. The high chemical shift for the coordinated proton in both compounds appears in the low-field region of the spectrum at around 19 ppm where there are no interfering signals providing us with an ideal NMR handle. Two peaks were seen: one at δ 19.49 ppm corresponding to $[\mathbf{1}\cdot H]^+$ and the other at δ 18.67 ppm corresponding to $[PS\cdot H]^+$. The size of the integrations for the NMR signals corresponding to $[\mathbf{1}\cdot H]^+$ and $[PS\cdot H]^+$ were averaged over 4 separate experiments. Using the average ratio of the integrations and the reported pK_a of PS (18.18 in CH_3CN) [37], the K_a of **1** was calculated to be $(1.50 \pm 0.06) \times 10^{18}$. The p$K_a$ is approximately equal to that of Proton Sponge® (18.2 ± 0.7 at 297 K in CD_3CN). Fig. 2 shows the relative intensities of the two signals for one of the NMR experiments.

The ESI mass spectrum of **1** in dichloromethane (and spiked with formic acid) is dominated by the $[M + H]^+$ peak as expected. There are no dimers, no coordination of sodium or potassium ions, and no doubly-charged species. The complete dominance of a single ionization pathway and the excellent signal intensity exhibited by **1** demonstrates its potential as an ESI tag (Fig. 3).

An energy dependent ESI (EDESI) experiment [40] was performed on **1** in which the cone voltage in the ESI source was raised in a stepwise manner to induce fragmentation. Spectra collected at each cone voltage are compiled into a two-dimensional contour plot featuring the mass-to-charge ratio on the horizontal axis and the cone voltage on the vertical axis. A single spectrum showing data summed over all collision voltages is shown above the contour plot. Using this format the fragmentation behaviour of **1** at all collision voltages can be viewed in one compact image. The resulting contour plot shows that the compound begins to fragment at a cone voltage of approximately 45 V with loss of a 15 Da fragment. This is consistent with loss of one methyl group as a neutral radical (Fig. 4): a fairly high-energy process consistent with the elevated cone voltage.

3.2. *Application of 1 as a chargeable tag for ESI-MS analysis*

To further test the suitability of **1** as an ESI probe, the η^6-coordinated chromium tricarbonyl complex of **1** was synthesized and studied by ESI-MS. Chromium hexacarbonyl in THF and dibutyl ether was reacted with **1** under reflux for 24 h [41]. After workup and recrystallization, yellow rod-like crystals were obtained and the crystal structure of chromium(**1**) tricarbonyl (**2**) was solved (Fig. 5).

The crystal structure shows the expected η^6-coordination of the arene ligand where the metal centre binds to the face of the arene that is pointing away from the nitrogen groups of the proton

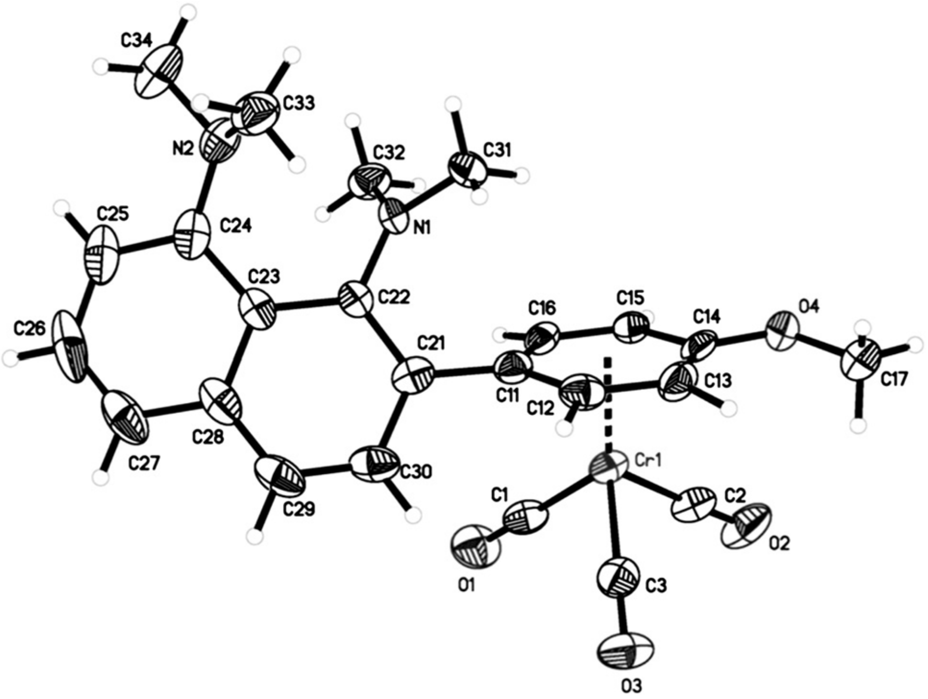


Fig. 5. Single crystal X-ray structure of chromium(**1**) tricarbonyl (**2**).

sponge moiety. This is ideal since the portion of the complex bearing the charge is far-removed from the reactive chromium centre and is unlikely to interfere with reactivity. The CO–Cr–CO angles of 88.1°, 88.7° and 90.3° are typical for a chromium tricarbonyl complex (87–92°) [42] indicating that steric perturbation of the complex due to addition of the chargeable group is minimal. Solution phase data for **2** is consistent with the crystal structure; the proton NMR spectrum displays signals consistent with η^6-coordination of the ligand and the IR spectrum displays CO stretches at 1964 cm^{-1} and 1890 cm^{-1} (compared to 1978 cm^{-1} and 1908 cm^{-1} for $Cr(\eta^6\text{-anisole})(CO)_3$) [43].

The ESI(+)-MS of **2** shows that the proton sponge tag behaves as expected. The spectrum is clean and dominated by the $[\mathbf{2} + H]^+$ signal (Fig. 6). No competing ionization processes are active. Some uncoordinated ligand is observed (*m*/*z* 321) perhaps indicating that the anisole ligand is labile in solution or that the arene–Cr interaction is relatively weak and some fragmentation of the compound occurs during the ESI process.

Further information regarding the structure and reactivity of **2** may be obtained through an EDESI experiment. In this case the experiment reveals that there is a threshold cone voltage of approximately 27 V at which all three CO groups are lost simultaneously (Fig. 7). While perhaps unintuitive, these results are consistent with earlier EI-MS studies by Dyson and McGrady in which the dominant fragmentation pathway for a number of similar chromium carbonyl complexes was concerted loss of all three carbonyl groups [44]. They also noted much smaller peaks corresponding to the di- and mono-carbonyl species, and on closer examination these peaks are observable in our spectrum as well: $[Cr(\mathbf{1})(CO)_2 + H]^+ = m/z$ 429 and $[Cr(\mathbf{1})(CO) + H]^+ = m/z$ 401. Since the fragmentation we observed matches with that found in the literature, we are further convinced that **1** is a suitable ESI-active probe.

At higher cone voltages (>40 V) decomposition of the ligand occurs with loss of methane (16 Da). In an attempt to further understand this decomposition process CID experiments were performed on $[\mathbf{2} + H]^+$ and deuterium labelled $[\mathbf{2} + D]^+$. Loss of 16 Da is observed as the major decomposition pathway (after CO dissociation) in the presence *and* in the absence of deuterons (Fig. 8). This outcome suggests that the fragmentation process does not involve loss of the acidic proton, or a loss of 17 Da would be expected when deuterium was incorporated. It is unclear what the mechanism of this fragmentation might be – there are three distinct types of methyl group present, and the propensity for C–H (and even C–N or C–C) activation by the Cr will be high following loss of all 3 CO ligands. Given the non-involvement of the deuteron, C–N and C–H activation of the closest NMe_2 group and reductive elimination of CH_4 seems the most probable pathway. What is important to note is that use of this ligand as an ESI tag is not advisable at high collision energies; however, this advice generally

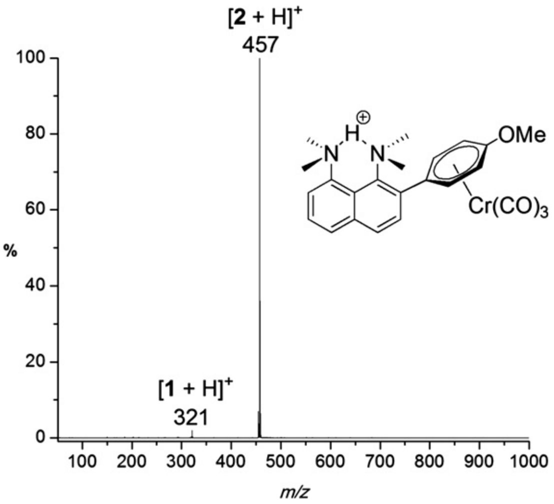


Fig. 6. ESI(+)-MS of **2** in methanol ($[\mathbf{2} + H]^+ = m/z$ 457).

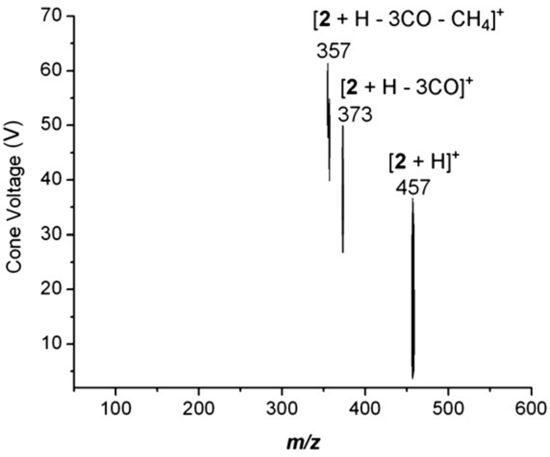


Fig. 7. EDESI(+)-MS of **2** in dichloromethane spiked with formic acid. Scan time = 2 s. CO dissociation (373 *m*/*z*) and ligand fragmentation (357 *m*/*z*) pathways are observed.

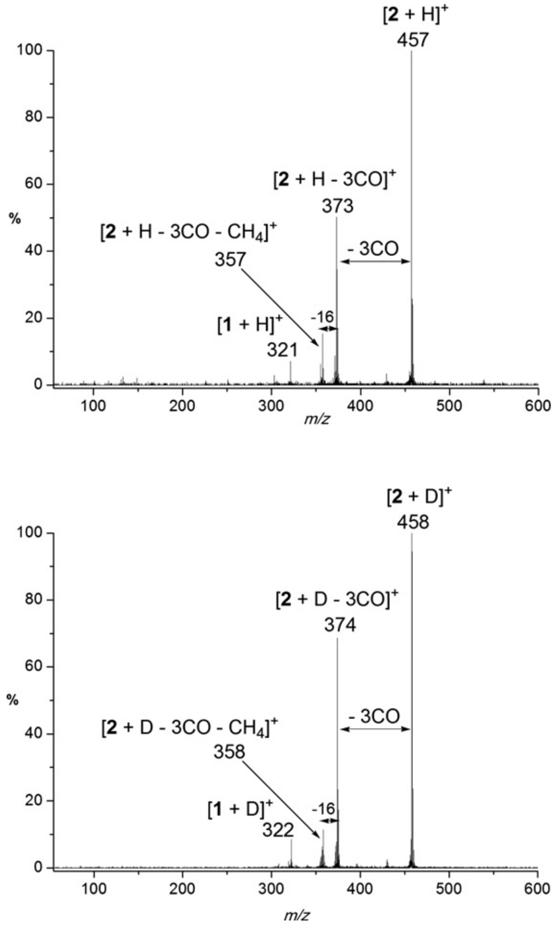


Fig. 8. (Top) ESI(+)-MS/MS of **2** in methanol spiked with formic acid. (Bottom) ESI(+)-MS/MS of **2** in d_1-methanol spiked with formic acid.

holds for most organometallic complexes due to their fragility compared to most organic molecules (in particular, organometallic complexes are susceptible to fragmentation thanks to loss of ligands as stable molecules, e.g. CO in this instance).

4. Conclusions

1,8-Bis(dimethylamino)-2-(4-methoxyphenyl)naphthalene (**1**) possesses all of the qualities of a useful chargeable tag for analysis by ESI-MS: the ligand readily coordinates to chromium in an η^6 fashion; the proton sponge moiety selectively and efficiently binds proton to become charged; and the charged site is far-removed from the active site of the metal so that reactivity of the chromium complex was found to be unaltered. Having proven its suitability as an ESI tag this ligand can now be employed in the ESI-MS study of organometallic reactions of neutral complexes with arene ancillary ligands.

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Appendix A. Supplementary material

Supplementary material associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.jorganchem.2012.07.009.

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