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Supporting Information

for

# Synthesis and Structural Characterization of Alkali Metal Arsinoamides

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# 1. NMR-Spectra

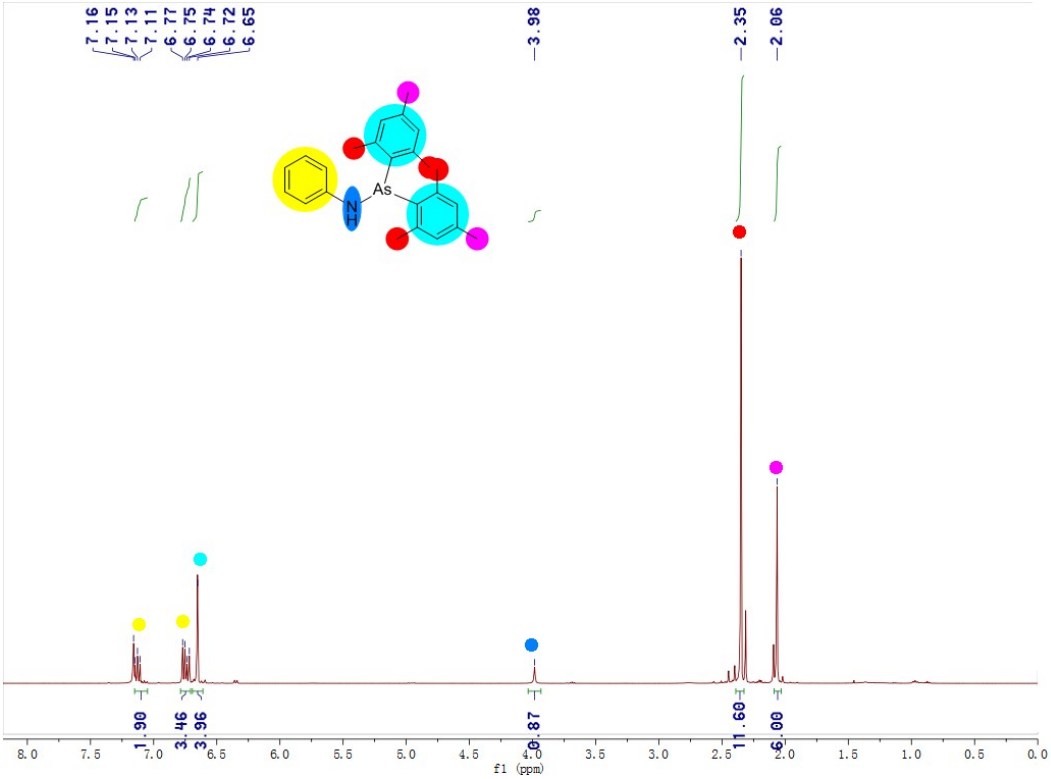


*Figure S1: $^{1}H$ NMR spectrum (400 MHz, 298K, $C_6D_6$) of **1**.*

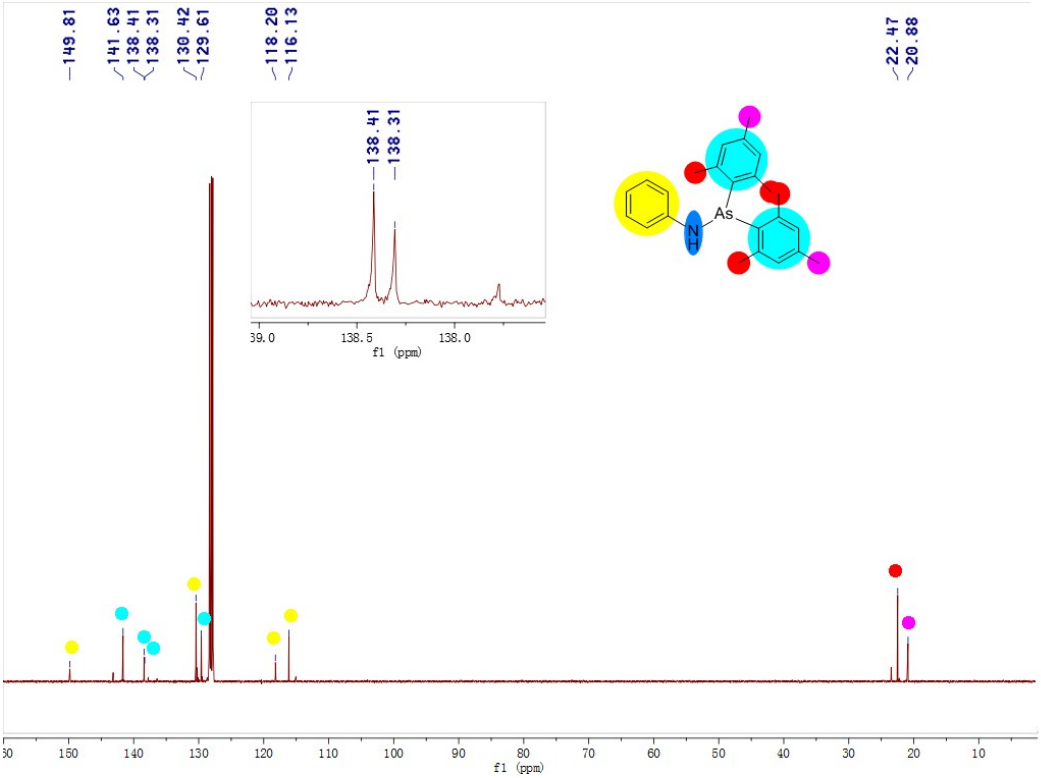


*Figure S2: $^{13}C\{^{1}H\}$ spectrum (100 MHz, 298K, $C_6D_6$) of **1**.*

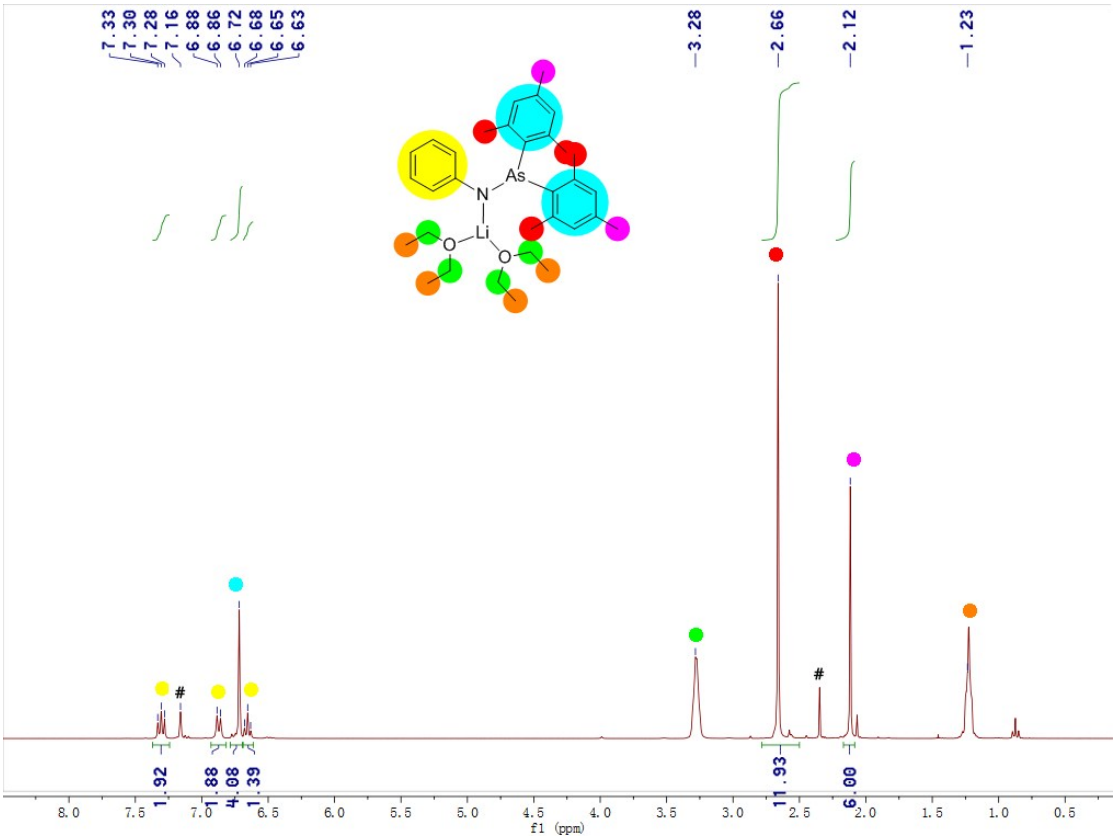


*Figure S3: $^{1}H$ NMR spectrum (300 MHz, 298K, $C_6D_6$) of **2** ("#" stands for solvent peak).*

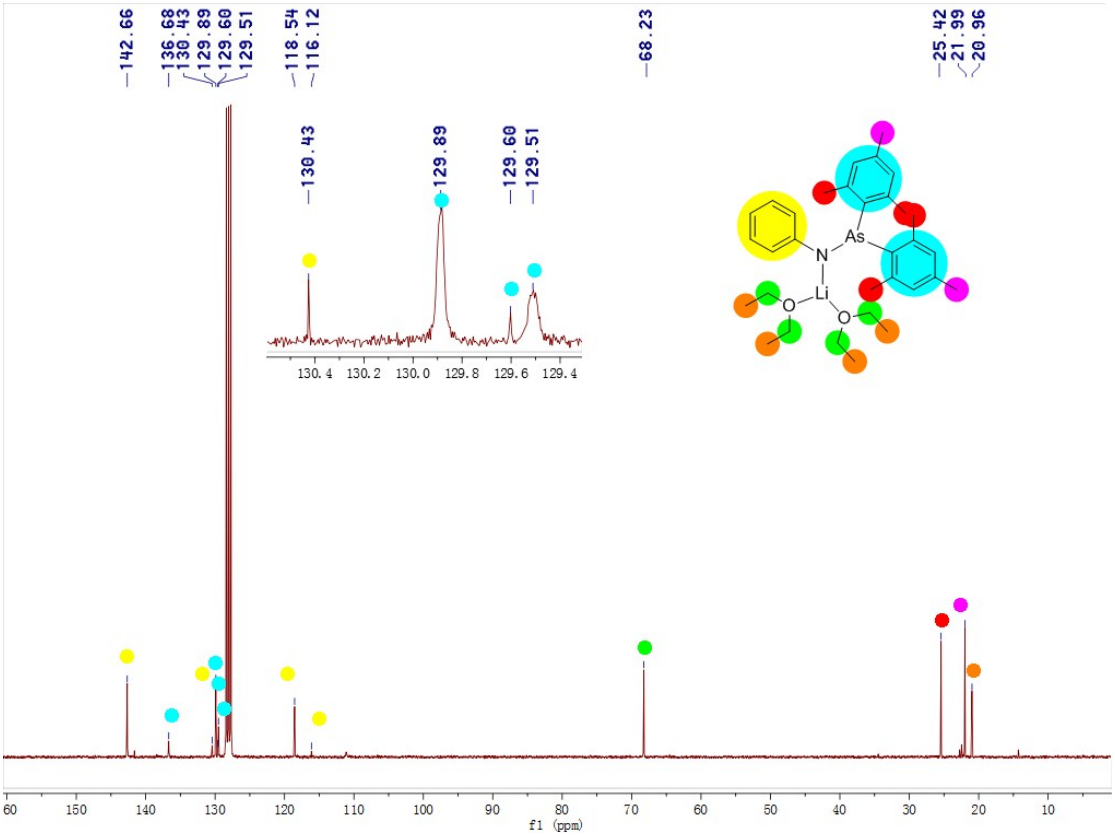


*Figure S4: $^{13}C\{^{1}H\}$ spectrum (75 MHz, 298K, $C_6D_6$) of **2**.*

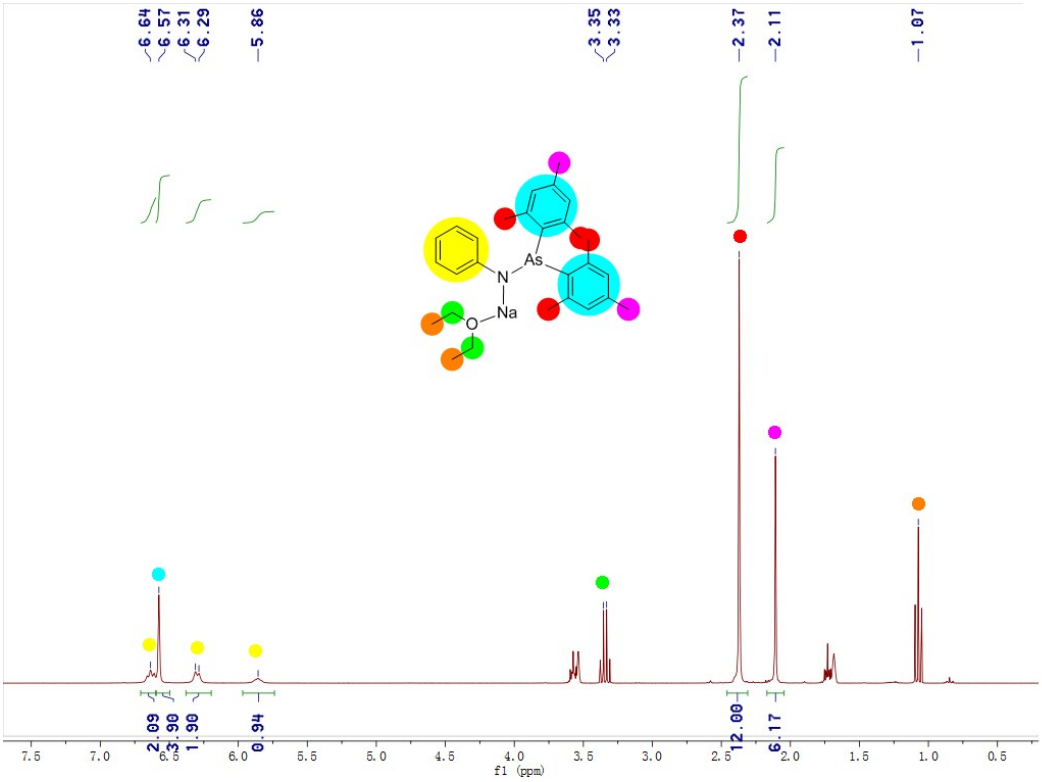


*Figure S5: $^{1}$H NMR spectrum (300 MHz, 298K, THF-$d_8$) of **3**.*

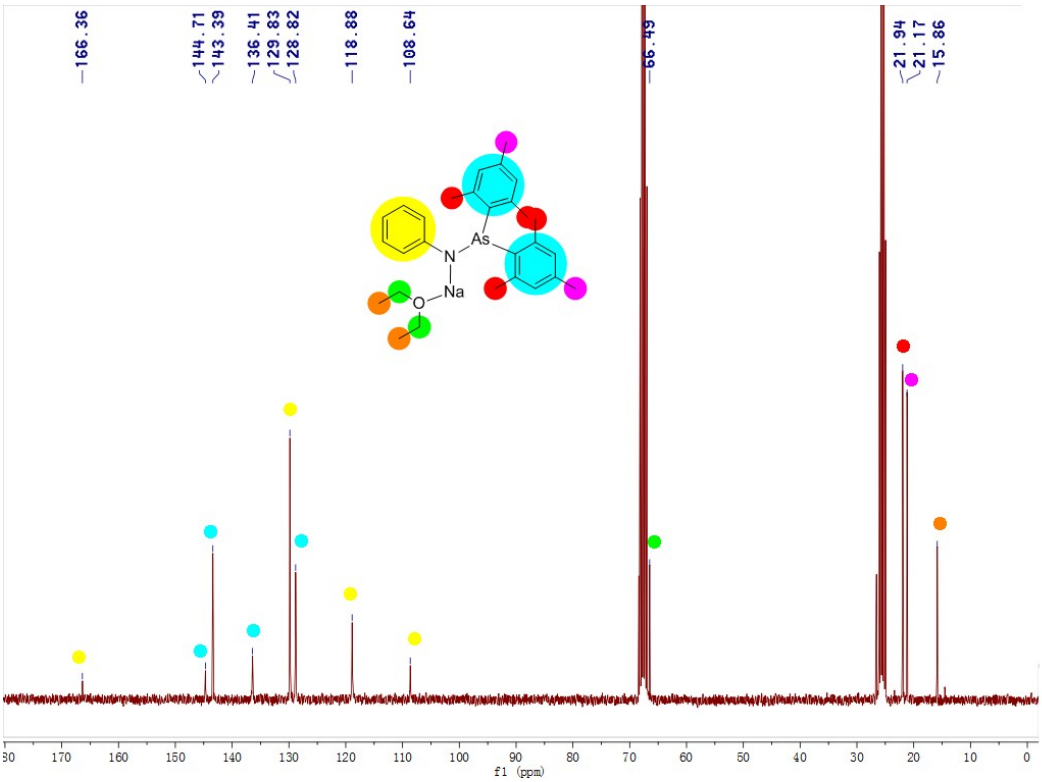


*Figure S6: $^{13}$C{$^{1}$H} spectrum (75 MHz, 298K, THF-$d_8$) of **3**.*

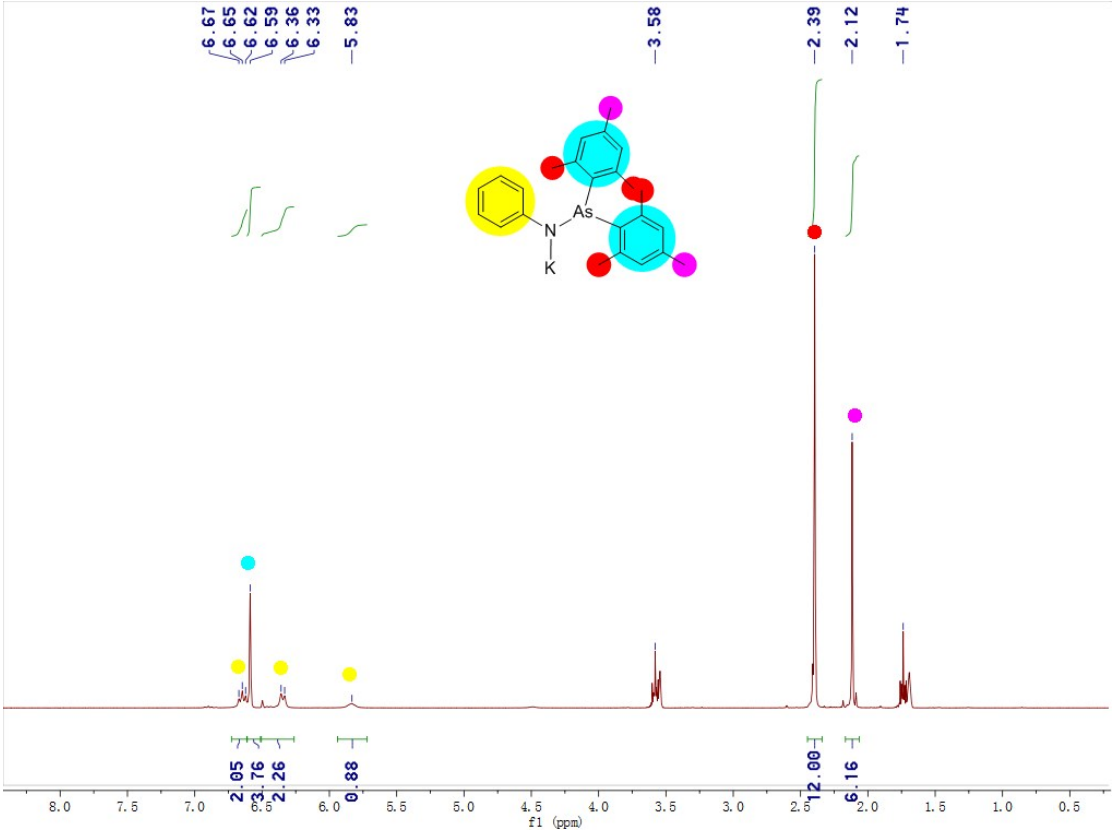


*Figure S7: $^{1}$H NMR spectrum (300 MHz, 298K, THF-$d_8$) of **4**.*

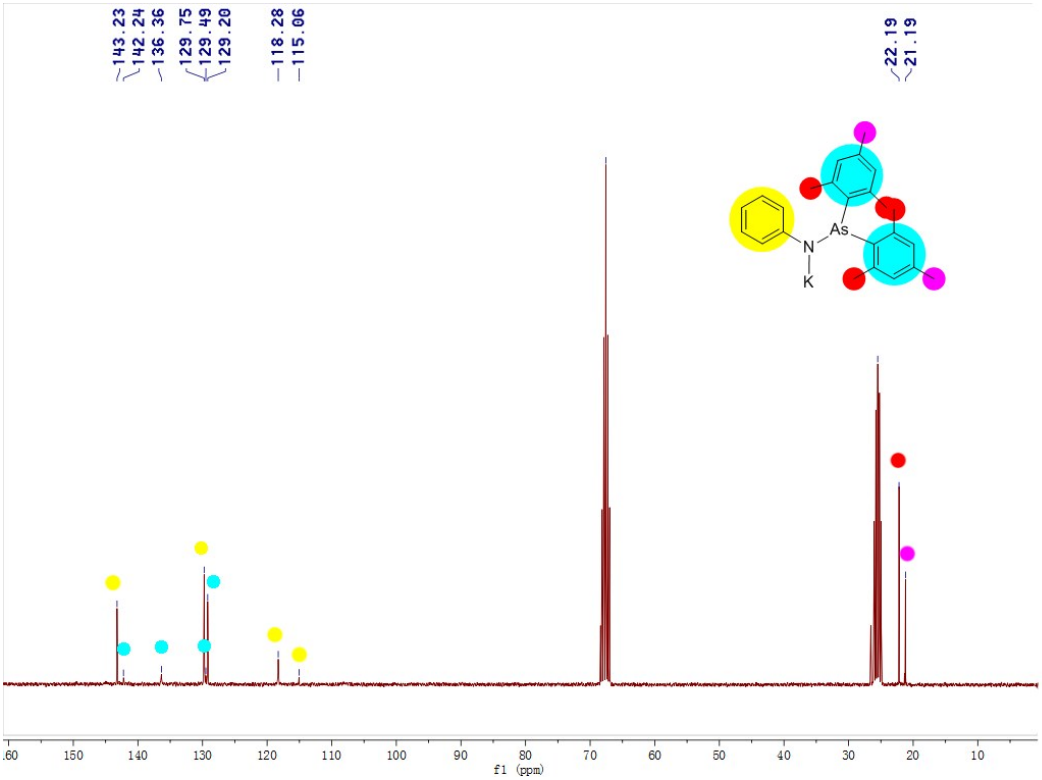


*Figure S8: $^{13}$C{$^{1}$H} spectrum (75 MHz, 298K, THF-$d_8$) of **4**.*

## 2. IR-Spectra

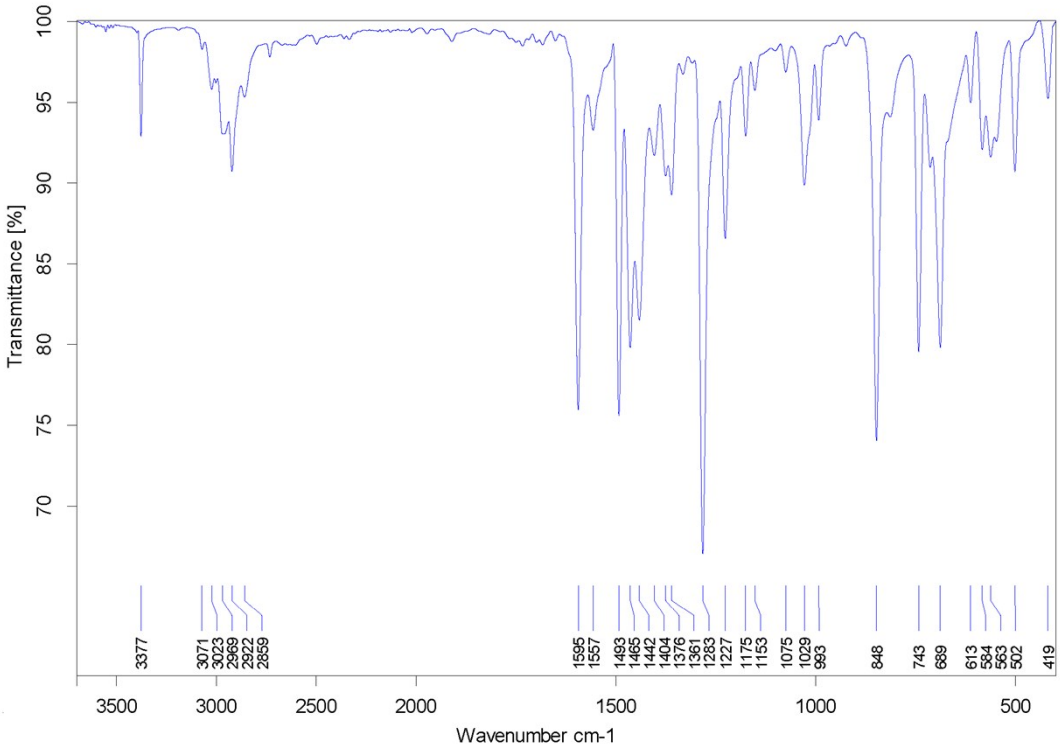


*Figure S9: **IR** spectrum of **1**.*

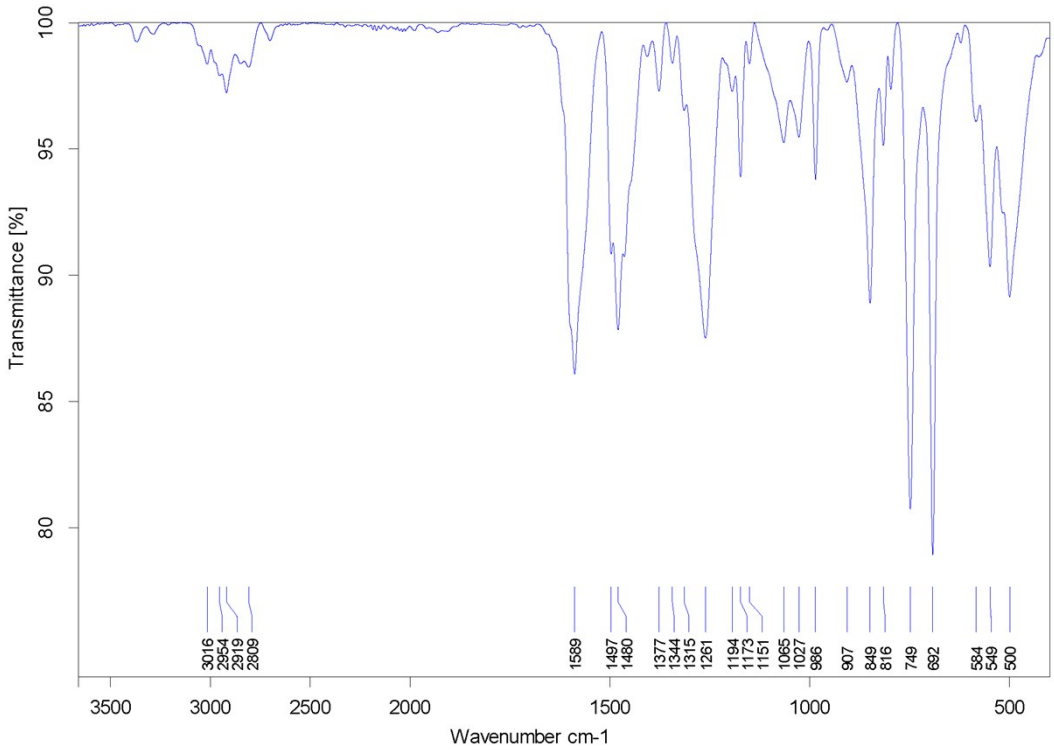


*Figure S10: **IR** spectrum of **2**.*

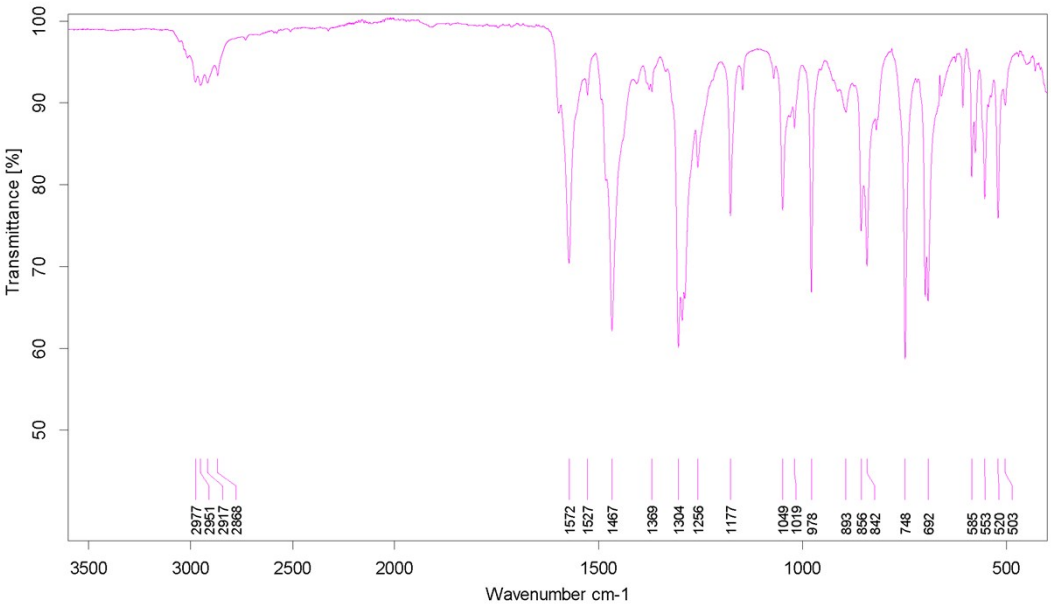


*Figure S11:* ***IR*** *spectrum of* ***3****.*

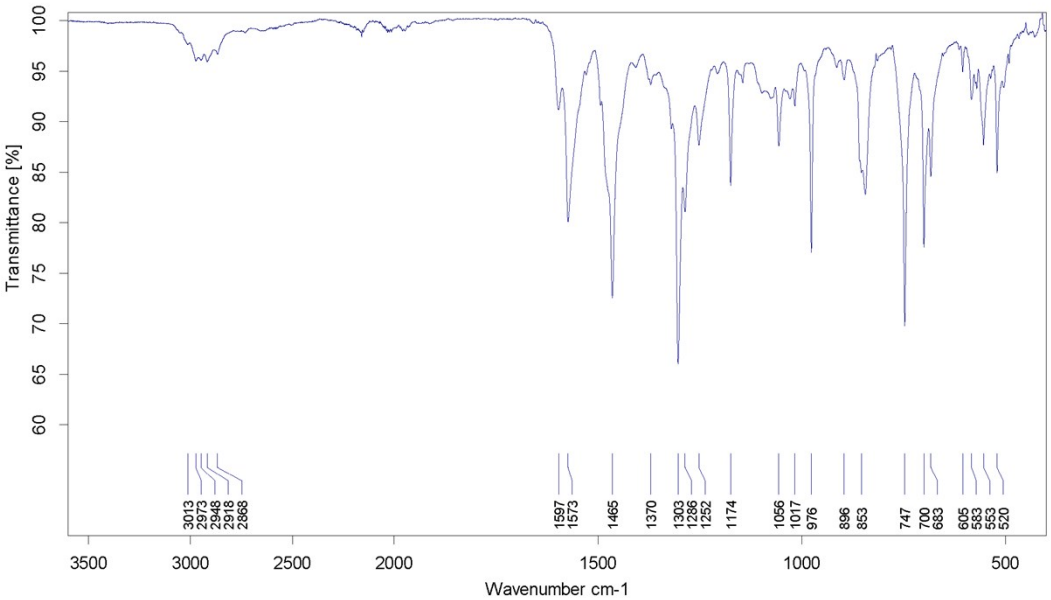


*Figure S12:* ***IR*** *spectrum of* ***4****.*

## 3. Crystallographic Investigations

A suitable crystal was covered in mineral oil (Aldrich) and mounted on a glass fiber or a mylar loop. The crystal was transferred directly to the cold stream of a STOE IPDS 2 diffractometer.

All structures were solved by using the program SHELXS/T[1, 2] and Olex2.[3] The remaining non-hydrogen atoms were located from successive difference Fourier map calculations. The refinements were carried out by using full-matrix least-squares techniques on $F^2$ by using the program SHELXL.[1, 2] In each case, the locations of the largest peaks in the final difference Fourier map calculations, as well as the magnitude of the residual electron densities, were of no chemical significance. Positional parameters, hydrogen atom parameters, thermal parameters, bond distances and angles have been deposited as supporting information.

Crystallographic data (excluding structure factors) for the structures reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as a supplementary publication no. CCDC-1586627-1586629. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB21EZ, UK (fax: (+(44)1223-336-033; email: deposit@ccdc.cam.ac.uk).

**Table S1.** Crystallographic Details for **2-4**.

| Compound reference | **2** | **3** | **4** |
|---|---|---|---|
| Chemical formula | $0.6(C_{32}H_{47}AsLiNO_2)/$ $0.4(C_{32}H_{45}AsLiNO_2)$ | $C_{56}H_{74}As_2N_2Na_2O_2$ | $C_{56}H_{70}As_2K_2N_2O_2$ |
| Formula Mass | 558.76 (= 0.6• 559,59 + 0.4•557,58) | 1002.99 | 1031.18 |
| Crystal system | monoclinic | triclinic | triclinic |
| Space group | $P2_1$ | $P$-1 | $P$-1 |
| $a$/Å | 9.7629(4) | 10.4020(6) | 9.7359(5) |
| $b$/Å | 16.3472(4) | 10.5518(6) | 10.7675(6) |
| $c$/Å | 10.4418(4) | 13.2663(8) | 14.0129(7) |
| $\alpha$/° | 90 | 82.314(5) | 67.450(4) |
| $\beta$/° | 109.809(3) | 75.737(5) | 81.984(4) |
| $\gamma$/° | 90 | 69.727(4) | 76.104(4) |
| Unit cell volume/Å$^3$ | 1567.86(10) | 1321.93(14) | 1315.14(13) |
| Temperature/K | 150 | 200 | 210 |
| No. of formula units per unit cell, $Z$ | 2 | 1 | 1 |
| Radiation type | MoKα | MoKα | MoKα |
| Absorption coefficient, $\mu$/mm$^{-1}$ | 1.11 | 1.321 | 1.47 |
| No. of reflections measured | 35999 | 12326 | 13130 |
| No. of independent reflections | 8466 | 7012 | 7205 |
| $R_{int}$ | 0.0786 | 0.0459 | 0.0378 |
| Final $R_1$ values ($I > 2\sigma(I)$) | 0.0631 | 0.0460 | 0.0496 |
| Final $wR(F^2)$ values ($I > 2\sigma(I)$) | 0.1519 | 0.1098 | 0.1174 |
| Final $R_1$ values (all data) | 0.0873 | 0.0841 | 0.0805 |
| Final $wR(F^2)$ values (all data) | 0.1599 | 0.1249 | 0.1353 |
| Goodness of fit on $F^2$ | 0.937 | 1.042 | 1.028 |
| Flack parameter | 0.01(2) | | |

**ORTEP-Plots of solid-state structures**

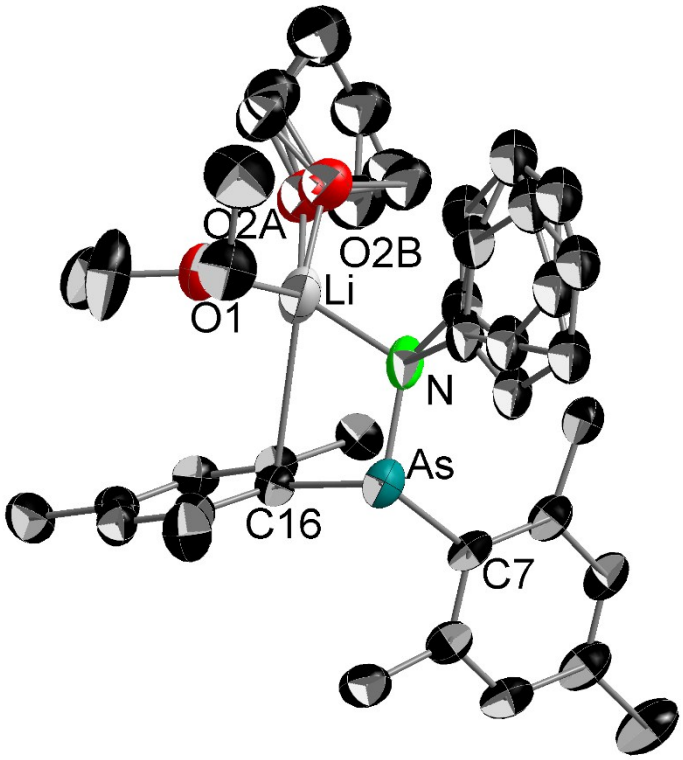


*Figure S13: Molecular structure of **2** in the solid-state. Hydrogen atoms are omitted for clarity. Ellipsoids displayed at 50% probability. Note: The position of one ether molecule (O2) is about 40% occupied by a THF molecule.*

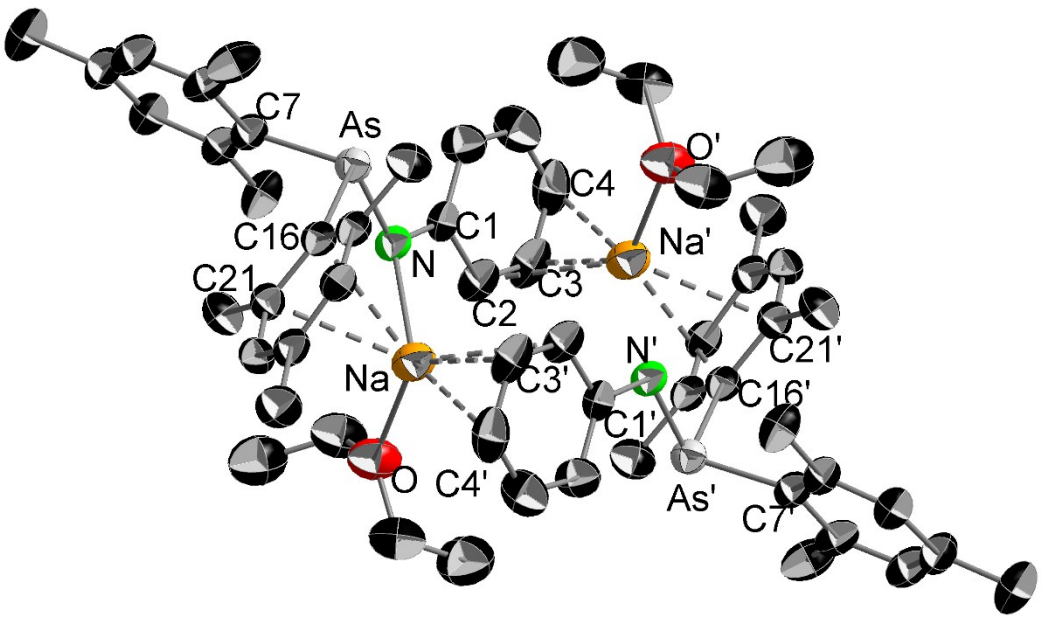


*Figure S14: Molecular structure of **3** in the solid-state. Hydrogen atoms are omitted for clarity. Ellipsoids displayed at 50% probability.*

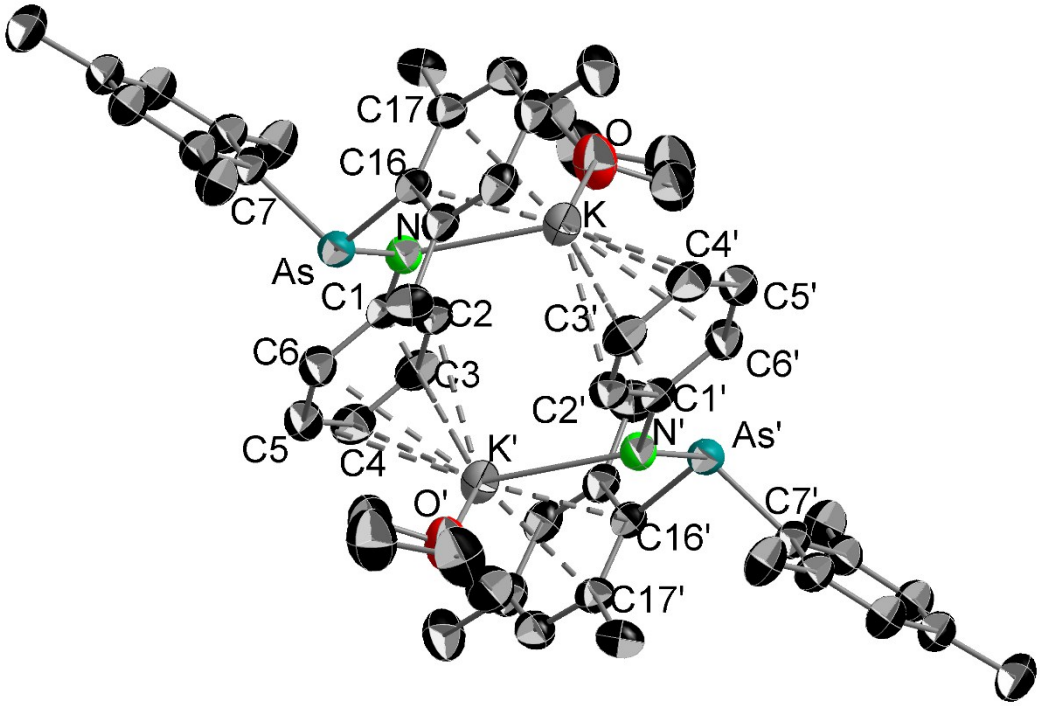


*Figure S15: Molecular structure of **4** in the solid-state. Hydrogen atoms are omitted for clarity. Ellipsoids displayed at 50% probability.*

# 4. References


1. G. Sheldrick, *Acta Crystallogr. Sect. A*, 2008, **64**, 112-122.
2. G. Sheldrick, *Acta Crystallogr. Sect. C*, 2015, **71**, 3-8.
3. O. V. Dolomanov, L. J. Bourhis, R. J. Gildea, J. A. K. Howard and H. Puschmann, *J. Appl. Crystallogr.*, 2009, **42**, 339-341.