Supplementary information A recipe for new organometallic polymers and oligomers? Synthesis and structures of oligo- and polymeric arrangements of P-S anions

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Experimental

All operations were carried out in an atmosphere of purified dinitrogen. Solvents were dried and freshly distilled prior to use. P_2S_5 and Davy's reagent were purchased from Aldrich. AgS^iPr was prepared according to a published procedure.[1]

1: 0.18 mg (1.00 mmol) AgS^iPr and 0.40 g (1.00 mmol) Dppe were dissolved in 8 mL THF. Separately, a yellow suspension of 0.22 g (0.50 mmol) Davy's reagent in 4 mL of THF is prepared and added to the solution of AgS^iPr and Dppe. The reaction is stirred for 2 hours and filtered. Crystals can be obtained by careful dropwise addition of diethylether to the stirring reaction mixture until a cloudy precipitate starts to form. The mixture is then stored at room temperature for three weeks before crystals of **1** can be isolated together with a colourless precipitate (30 mg, 10% based on AgS^iPr supplied) (Found C, 41.8; H, 3.6. $C_{255}H_{263}Ag_{24}P_{17}S_{33}$ requires C, 40.9; H, 3.5); δ_P(161.975 MHz, $CDCl_3$, 25°C, H_3PO_4) 132.8, 125.3, 115.5 ($[PS_4]^{3-}$), 103.3 (S_3PS^tBu), 44.5, 32.6 ($Ph_2P(S)$-), 0 br (Ph_2P).

2: A mixture of P_2S_5 222 mg (1.00 mmol) and NaS^tBu 448 mg (4.00 mmol) was dissolved in 12 mL DME. The mixture was stirred overnight and the filtrate was layered with 60 mL hexane. Storage of this solution at room temperature for one week produced colorless crystals of **2** (0.48 g, 68% based on NaS^tBu supplied); found C, 25.7; H, 4.9. $C_{14}H_{33}Na_4O_3P_2S_8$ (**2**-0.5DME) requires C, 25.5; H, 5.0; m.p. 120°C (decomposition); δ_H(400 MHz, [D_8]THF, 25°C, TMS) 3.46 (s, 4H, CH_2O), 3.31 (s, 6H, OCH_3), 1.63 (s, 9H, S^{t}Bu); δ_C(100 MHz, [D_8]THF, 25°C) 71.76 (O-CH_2), 57.98 (O-CH_3), 49.27, 49.21 (S-C), 31.52, 31.48 (CH_3); δ_P(161.975 MHz, [D_8]THF, 25°C,H_3PO_4) 93.3 (S_3PS^tBu); ν_{max}(NaCl, Nujol)/cm^{-1} 1624s, 1245m, 1169s, 1034s, 855s, 602s.

CCDC no. 623841 and 623842 contain the supplementary crystallographic data for this paper. These data can be obtained free of charge atwww.ccdc.cam.ac.uk/products/csd/request [or from the Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge CB2 1EZ, UK; fax: (internat.) +44 1223/336 033; E-mail: deposit@ccdc.cam.ac.uk].

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† Electronic Supplementary Information (ESI) available: [details of any supplementary information available should be included here]. See http://dx.doi.org/10.1039/b000000x/

References

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