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Preliminary communication

The preparation of a series of ring-linked derivatives of $[Fe_2(\eta\text{-}C_5H_5)_2(CO)_2(\mu\text{-}CO)_2]$ starting from $[Fe_2\{\eta^5,\eta^5\text{-}C_5H_4CH(NMe_3)CH(NMe_2)C_5H_4\}(CO)_2(\mu\text{-}CO)_2]^+$ salts

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Abstract

The salts $[Fe_2\{\eta^5,\eta^5\text{-}C_5H_4CH\{NMe_3)CH(NMe_2)C_5H_4\}(CO)_2(\mu\text{-}CO)_2][X]$ (X = I or SO_3CF_3) are the synthetic precursors to a wide range of $[Fe_2(\eta\text{-}C_5H_5)_2(CO)_2(\mu\text{-}CO)_2]$ derivatives in which the two cyclopentadienyl ligands are joined by a two-carbon bridge.

$[Fe_2\{\eta^5,\eta^5\text{-}C_5H_4CH(NMe_3)CH(NMe_2)C_5H_4\}(CO)_2(\mu\text{-}CO)_2][X]$ salts are readily prepared by the methylation of either *R,S* or *R,R/S,S* isomers of $[Fe_2\{\eta^5,\eta^5\text{-}C_5H_4CH(NMe_2)CH(NMe_2)C_5H_4\}(CO)_2(\mu\text{-}CO)_2]$ [1].

Treatment of a solution of these salts in acetonitrile with a slight excess of $[PhCH_2NMe_3]OH$ (Triton-B) in methanol or water gives an almost quantitative yield of the enamine $[Fe_2\{\eta^5,\eta^5\text{-}C_5H_4C(H)C(NMe_2)C_5H_4\}(CO)_2(\mu\text{-}CO)_2]$ (dec. 260 °C), contaminated with traces of $[Fe_2\{\eta^5,\eta^5\text{-}C_5H_4CH(NMe_2)CH(NMe_2)C_5H_4\}(CO)_2(\mu\text{-}CO)_2]$.

Although $[Fe_2\{\eta^5,\eta^5\text{-}C_5H_4C(H)C(NMe_2)C_5H_4\}(CO)_2(\mu\text{-}CO)_2]$ could be purified by recrystallization, attempts to do this by chromatography on alumina resulted in its acid-catalysed hydrolysis to $[Fe_2\{\eta^5,\eta^5\text{-}C_5H_4CH_2C(O)C_5H_4\}(CO)_2(\mu\text{-}CO)_2]$ (m.p. 213–215 °C). This undergoes many of the usual reactions of a ketone [2]; e.g. $[NH_3OH]Cl$ converts it into an oxime $[Fe_2\{\eta^5,\eta^5\text{-}C_5H_4CH_2C(NOH)C_5H_4\}(CO)_2(\mu\text{-}CO)_2]$ (m.p. 237–239 °C), and it is reduced by $LiAlH_4$ to a secondary alcohol, $[Fe_2\{\eta^5,\eta^5\text{-}C_5H_4CH_2CH(OH)C_5H_4\}(CO)_2(\mu\text{-}CO)_2]$ (m.p. 188–191 °C).

$LiAlH_4$ reduces the salts $[Fe_2\{\eta^5,\eta^5\text{-}C_5H_4CH(NMe_3)CH(NMe_2)C_5H_4\}(CO)_2(\mu\text{-}CO)_2][X]$ (X = I or SO_3CF_3) quantitatively to $[Fe_2\{\eta^5,\eta^5\text{-}C_5H_4CH_2CH(NMe_2)C_5H_4\}(CO)_2(\mu\text{-}CO)_2]$ (dec. 245 °C). This in turn may be methylated by $MeOSO_2CF_3$ to give $[Fe_2\{\eta^5,\eta^5\text{-}C_5H_4CH_2CH(NMe_3)C_5H_4\}(CO)_2(\mu\text{-}CO)_2][SO_3CF_3]$ (dec. 202 °C).

Analytically pure samples of all products were isolated, and characterised by IR and NMR spectroscopy. The $Fe_2(\eta\text{-dienyl})_2(CO)_2(\mu\text{-}CO)_2$ part of the molecule is

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unchanged by the above reactions; e.g. for $[Fe_2\{\eta^5,\eta^5\text{-}C_5H_4CH_2C(O)C_5H_4\}\text{-}(CO)_2(\mu\text{-}CO)_2]$ $\nu(CO)$ (carbon disulphide solution; relative peak heights in parentheses): 2005(10), 1968(2), 1819(0.4), 1792(6.5), 1688(1.5; ketone) cm^{-1}.

References

1 A.R. Manning, A. McCabe, and P. Soye. in preparation.

2 H.T. Openshaw, A Laboratory Manual of Qualitative Organic Analysis, 2nd edition, Cambridge University Press, London, England, 1951.