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PREPARATION OF NEW GRAPHITE INTERCALATION COMPOUNDS IN ANHYDROUS HYDROGEN FLUORIDE

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Several new graphite intercalation compounds (GIC) have been obtained by preparing the intercalant in situ in AHF solutions and reacting these with graphite. The products were subjected to exhaustive chemical analyses and attempts were made to achieve mass balances between the starting materials and the final GIC products. The following systems were studied:

$C + I_2O_5\ (KIO_3) + AHF \longrightarrow$ no reaction (1)

$C + KReO_4 + AHF \longrightarrow$ no reaction (2)

$C + H_5IO_6\ (NaIO_4) + AHF \longrightarrow C_{\sim 10}IO_{\sim 2.7}F_{\sim 4.7}$ (3)
(Stage I; $d_0 = 8.30$Å ; Stage II; $d = 11.66$Å)

$C + V_2O_5\ (NaVO_3) + AHF \longrightarrow C_{18.9 \pm 0.2}VOF_{3.4 \pm 0.1}$ (4)
(Stage III; $d = 14.71$Å ; $d_0 = 8.01$Å)

$C + CrO_3\ (K_2Cr_2O_7) + AHF \longrightarrow C_{8.9 \pm 0.5}CrO_2F_2$ (5)
(Stage I; $d_0 = 7.95$Å)

$C + KMnO_4\ (MnO_3F) + AHF \longrightarrow C_{8-9}Mn_{2 \pm 0.5}F_{4-5}$ (6)
(Stage II; $d = 11.22$Å ; $d_0 = 7.87$Å)

For reaction (3) a combination of oxidation power and F/I analyses indicates that the intercalant consists of mixtures of heptavalent (probably $IO_2F_4^-$) and pentavalent ($IO_2F_2^-$ or IOF_4^-) species. In reaction (6) a paramagnetic GIC is obtained with possibly binuclear intercalate Mn species. The inclusion of HF in the products cannot be ruled out except for reaction (5).

We conclude that the prevalent practice of quoting stoichiometries based only on weight uptakes may be misleading as this does not take into account the possible presence of reduced species.