

E2-33 Extractive spectrophotometric determination of Tungsten [VI] by ternary complex formation

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Tungsten [VI] reacts with N-benzoylphenylhydroxylamine [NBPHA] in 2M HCl acid medium to give a colourless complex, which can be completely extracted into dichloromethane.

A synergistic effect was observed if the extraction was carried out in the presence of ethylacetoacetate [EAA]. On treatment of the dichloromethane extract with phenylfluorone in dimethylformamide and acetone, an intensely coloured complex was formed. This complex exhibits an absorption maximum at 492 nm and obeys Beer's law upto 8.5 mgdm^{-3} of tungsten [VI]. The molar absorptivity at the optimum conditions is $2.4 \times 10^4 \text{ dm}^3 \text{ mol}^{-1} \text{ cm}^{-1}$.

From Job's method, it was confirmed that the stoichiometric ratio of tungsten [VI] to NBPHA is 1:1. However, an excess of EAA is needed to show a synergistic effect in this system. From the infra-red spectroscopic data it is possible to confirm the existence of W[VI]-O and W [VI]-N bonding in the extracted complex.

The interference from molybdenum [VI], vanadium [IV], vanadium [V], zirconium [IV], titanium [IV], iron [II], iron [III], aluminium [III], fluorides and phosphates was studied. The system can tolerate a large excess of cations and anions such as, iron [II], iron [III], aluminium [III], fluorides and phosphates but critical interferences from molybdenum [VI], vanadium [IV], vanadium [V], zirconium [IV], titanium [IV] have been observed. This method was successfully applied to determine tungsten [VI] in synthetic mixtures.