Four new azo dyes, L-1, L-2, L-3, and L-4, were prepared by linking benzaldehyde p-aminobenzoylhydrazone (3) and p-hydroxybenzaldehyde p-aminobenzoylhydrazone (4) to barbiturie acid and 1,3-dimethylbarbituric acid through diazo-coupling reactions. Reactions of the azo-dyes with copper chloride and bidentate ligand, 1,10-phenanthroline, produced mixed-ligand dinuclear complexes with general stoichiometry (Cu2L(phen)(2)Cl2 (7, 8, 9, and 10). The structures of both azo dyes and their complexes were identified by elemental analyses, FT-IR, H-1-NMR, UV-VIS, magnetic susceptibility, and mass spectral data.