CHAPTER V

PRELIMINARY STUDIES OF METALLOCHROMIC PROPERTY OF 2,7-BIS[(2-HYDROXY-5-NITROPHENYL)AZO]-1,8DIHYDROXY-3,6-NAPHTHALENEDISULFONIC ACID (5N-DYE)

5.1 Experiment on Metallochromic Property of 5N-Dye

Procedure:

Preliminary studies of metallochromic property of the dye at pH 2-12 were carried out. Two series of the test tubes containing 1.00 mL of deionized water, 2 drops of the 0.5% 5N-dye solution and 2 drops of pH adjusting solution were placed on a test tube rack. To one series of the test tubes, was added 2 drops each, the solution of 10⁻² F metal ions. The solution in each series was adjusted to pH 2, 4, 7, 10, 11.5, and 12 by pH adjusting solution. Color change due to the complex formation of metal ion with dye was observed and compared with the dye alone, 3 drops of 10⁻² F EDTA was added into the test tube of metal-dye complex and any color change was then recorded as shown in Table 5.1.

Table 5.1 Visual color change studies of the 5N-dye

| pН | | | | | | |
|------------|-------------------|-----------------------------|-------------------|-------------------|-------------------|-------------------|
| Metal | 2 | 4 | 7 | 10 | 11.5 | 12 |
| Dye(alone) | P | P | g | g | g | b-g |
| Bi(III) | b ^y | $\mathfrak{b}^{\mathrm{y}}$ | g-b ^z | - | - | - |
| Pb(II) | v-b ^x | v-b ^x | b ^x | ppt | ppt | - |
| Na(I) | - | | | <u>-</u> | <u>-</u> | - |
| Ni(II) | | v-b ^y | b ^z | - | - | - |
| Cd(II) | - | - 9 | b ^x | g-b ^y | g-b ^y | ppt |
| Mn(II) | v-b* | v-b ^x | b ^x | ppt | ppt | ppt |
| Fe(III) | v-b ^x | br ^x | gray ^x | ppt | - | - |
| Fe(II) | - | v ^x | gray ^x | gr-g ^x | gr-g ^x | gr-g ^x |
| Ag(I) | - | // / = | <u> </u> | \\ <u>-</u> | - | ppt |
| Sn(II) | • | | | | • | • |
| Co(II) | (- // | v ^x | + | + | + | + |
| Zr(II) | b ^x | p _x | g-b ^x | ppt | - | - |
| Cu(II) | b ^y | g-b ^y | b ^x | b-g ^x | b-g ^x | + |
| Zn(II) | + | + | b ^x | b-g ^y | b-g ^y | b-g ^y |
| Y(III) | ppt | ppt | b-g ^x | ppt | ppt | + |
| Cs(III) | + | b ^x | b-g ^x | + | + | + |
| Mg(II) | ij- | • | b ^x | -(1) | <u>.</u> | ppt |
| Ti(VI) | ppt | v-b ^x | g-b ^x | ppt | ppt | ppt |
| Pd(II) | b ^x | b ^x | g-b ^x | | 6. | <u>-</u> |
| Ba(II) | ppt | b ^x | g-b* | 9[] | l d - | - |
| Al(III) | b ^x | b ^x | g-b ^x | b-g ^x | b-g ^x | <u>-</u> |

Note 1

- = no color change, + = little color change, p = purple, g = green
- b = blue, gr = gray, br = brown, v-b = violet-blue, gr-g = gray green

g-b = greenish-blue, b-g = blueish-green, ppt. = precipitate

- x = no color change after the EDTA addition (irreversible)
- y = color change to original dye color at the same pH after the EDTA addition (reversible)
- z = color change but not same original dye color at the same pH after the EDTA addition
- = color changed to pale shade due to decomposition

5.2 Possible Application of 5N-Dye as Visual Indicator in a Complexometric Titration of Bismuth(III)

From the previous result in table showed that it might be possible to use 5N-dye as a visual indicator in a direct EDTA titration for the determination of bismuth(III). Therefore, an experiment was carried out and the result compared to those with xylenol orange as indicator.

Procedure

Dilute 25.0 mL of the bismuth-ion solution with deionized water to about 150 mL. Adjust, if necessary the pH to 2.00-3.00 by the cautious addition of dilute aqueous ammonia (use pH meter). Add 3-4 drops of the indicator solution titrated with standard EDTA until the red color commences to fade and then introduce the titrant more slowly. At the end point the color changes sharply from red to orange yellow when using xylenol orange as indicator. For 5N-dye, at the end point the color changes from blue to purple.

Table 5.2 Complexometric titration of bismuth(III) with xylenol orange and 5N-dye as indicator

| Indicator | Volume of EDTA 0.05 M | | | Color change at | Concentration of Bi(III) | |
|----------------|--------------------------|-------|-------|-----------------|--------------------------|--|
| | 1 | 2 | 3 | the end point | found (M) | |
| Xylenol orange | 25.05 | 25.00 | 25.00 | red orange | 0.0500 | |
| 5N-dye | 24.80 | 24.80 | 24.80 | blue> purple | 0.0496 | |

From Table 5.2, it shows that 5N-dye can be used as visual indicator for the direct complexometric EDTA titration of bismuth(III) though the final end point is not as sharp as those obtained for xylenol orange.