Qualitative Organic Analysis Lab

Detection of extra elements, nitrogen (N), sulfur (S) and halogens (X), in the sodium fusion extract (SFE) is a key step in the systematic analysis of organic compounds.

While nitrogen and sulfur are easily detected by usual tests (Box 1), a combination with halogens often proves difficult. This is so because the test for halogens in presence of N and/or S involves boiling the fusion extract with HNO₃ till N and S are expelled as HCN and H₂S. However, complete removal is seldom achieved.

Box 1

Nitrogen is tested by boiling SFE (pH-13) with solid ferrous sulfate¹ and acidifying with dilute H₂SO₄. Prussian blue colouration indicates nitrogen.

In presence of sulfur, the SFE develops purple colouration with sodium nitroprusside.

This difficulty is overcome thus: nitrogen and sulfur are tested in the usual manner. In the AgNO₃ test for halogens, the alkaline solidum fusion extract is treated with AgNO₃ to precipitate Ag₂S, AgCN and AgX together. The precipitate is separated, washed with distilled water and boiled with concentrated HNO₃. If it dissolves, halogen is absent. In case the precipitate survives, it is indeed AgX! Identity of X is then established by the layer test (Box 2).

Box 2

Layer test is best performed by acidifying the fusion extract with dilute HNO₃, forming a lower organic layer with CCl₄ or CHCl₃ and oxidising X in the fusion extract by 1%aq.KMnO₄ solution. Vigorous shaking then liberates the molecular halogen, Br₂ or I₂, which is taken up by the organic layer, colouring it orange or purple. If a precipitate survives the treatment of concentrated HNO₃ and fails to give a positive layer test, it must be Cl!

¹ Ferrous sulfate may be replaced by ferrous ammonium sulfate (Mohr's salt) which is about 5 times cheaper and serves the same purpose.

Suggested Reading