Notes

IDENTIFICATION OF PHENOL

An easily produced, yet little known, compound of phenol and hexamine (hexamethylenetetramine) can be used for the identification of phenol, either alone or in presence of the cresols.

The compound, which seems to have been first described by Moschatos and Tollens[1] in 1891, is considered to be composed of 3 molecules of phenol combined with 1 molecule of hexamine. This composition was confirmed by Harvey and Baekeland,[2] although Smith and Welch[3] considered it to be a 1 + 1 addition compound. Some account of the substance is given by Ellis.[4] The composition does not seem, however, to be of much importance where the test is concerned, as the formation of the needle-like crystals from dilute solution is so distinctive and can be readily observed. As the crystals decompose on melting at between 100° and 125° C, according to the solvent from which they are recrystallised, there is no sharp melting point that can be used for purposes of identification; but, in the author's opinion, the distinctive appearance and conditions of formation of the crystals render such extra identification unnecessary.

The compound is readily produced by adding a strong solution of hexamine to phenol saturated with water. If a solution of phenol in water is used, the formation of needle-like crystals can be observed. The reaction is best carried out on a watch-glass by adding 3 or 4 drops of saturated hexamine solution to 1 ml of phenol solution. By this method, phenol alone in aqueous solution can be identified at as low a concentration as 4 per cent. If crystals do not form immediately, the mixture is allowed to stand on the watch-glass for a while; crystals will then begin to form from the edge of the liquid as evaporation proceeds, 5 to 10 minutes being a sufficient time for the needle-like crystals to be observed, especially if a lens is used. Crystallisation can be hastened by scratching with a glass rod. As the crystals are fairly soluble in water, addition of an excess of hexamine solution must be avoided, as this increases the volume of water unduly and so prevents crystallisation.

The cresols also produce similar compounds, but by no means so readily as phenol itself, it being necessary to use the cresol layer of the cresol - water mixture, and even then the *o*-cresol compound is difficult to obtain. A saturated solution of the cresols does not yield crystals under the conditions described.

If, therefore, a mixture is being examined, and by odour and general reactions it is obviously phenolic in character, the presence of phenol can be demonstrated by the following procedure. The mixture is shaken with water and the phenolic layer is allowed to settle. Then to 1 ml of the aqueous layer are added 3 or 4 drops of saturated hexamine solution, the reaction being carried out on a watch-glass as described previously. The formation of the needle-like crystals can be taken as evidence of the presence of phenol. The author has found that the presence of up to a total of about 40 per cent. of all or any of the cresols before the sample is shaken with water does not interfere with the test.

As nearly all the usual tests described for phenol are responded to similarly by the cresols, the test here described should be useful, as it is simple to perform and is specific for phenol.

Identification of formaldehyde

By the converse of the above reaction, solutions of formaldehyde as dilute as 4 per cent. can be identified, the required hexamine being formed in the solution.

About 0·05 g of phenol crystals are placed on a watch-glass, and just sufficient of a 1 + 1 mixture of ammonium hydroxide, sp.gr. 0·880, and water is added dropwise to dissolve the crystals. Up to about 10 drops of the suspected formaldehyde solution are then added. If the liquid becomes turbid owing to the formaldehyde reacting with the ammonium hydroxide, a drop or two more of ammonium hydroxide is added just to clear it. The solution is then allowed to stand for a few minutes, when the typical needle-like crystals are observed forming at the edges if formaldehyde is present.

As formaldehyde is the only aldehyde that yields the necessary hexamine, the test is specific.

References

1. Moschatos, H., and Tollens, B., *Ber.*, 1891, **24**, 695.
2. Harvey, M. T., and Baekeland, L. H., *J. Ind. Eng. Chem.*, 1921, **13**, 135.
3. Smith, L. H., and Welch, K. N., *J. Chem. Soc.*, 1934, 729.
4. Ellis, C., "Chemistry of the Synthetic Resins," Reinhold Publishing Corporation, New York, 1935, Volume 1, p. 308.

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First submitted, *June 30th*, 1953
Amended, *January 5th*, 1954