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A NOTE ON THE REMOVAL OF SILVER FROM AQUEOUS EXTRACTS OF PLANT MATERIAL

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Summary

A modification of the Johnson and Ulrich method, involving precipitation at pH 10·5, is given.

Some difficulty has been experienced with the complete removal of silver from the aqueous extracts of plant material using the method of Johnson and Ulrich (1950). Because of this, tests were performed to determine the pH value at which silver is completely precipitated from solution as the phosphate, using weights of silver sulphate and potassium dihydrogen phosphate present in the extraction procedure.

Eleven solutions (25 ml each) were each adjusted to different pH levels ranging from pH 3·5 to pH 7·0 and then the precipitated phosphate was removed by filtration through a Whatman No. 42 paper. To the clear filtrates, after acidification with nitric acid, hydrochloric acid was added and the precipitation of any silver chloride was noted. Filtrates from the solutions having pH greater than 5·8 showed no detectable silver chloride precipitate. Using the extraction procedure of Johnson and Ulrich, complete removal of the excess silver ions as the phosphate occurs at pH 5·8. Filtration difficulties were encountered in the removal of the precipitated silver phosphate. The solutions below pH 6 had to be repassed through the paper to obtain a clear filtrate. Filtration was rapid and efficient with the solutions at pH 6·5 and pH 7·0. In practice, it is advisable to precipitate silver phosphate from solution at about pH 7.

An investigation was made into the pH changes occurring during the extraction of the plant material and the subsequent removal of excess silver from the extract. The extraction procedure of Johnson and Ulrich was used in each test. A total of 16 extractions was made, using two different weights of silver sulphate. Eight of the extractions contained no plant material blanks, four contained 0·1 g plant material and the remaining four contained 0·2 g plant material. Half of the blank extractions and those containing plant material were filtered. The remainder of the extractions were not filtered. The composition of each of the 16 extracts together with their corresponding pH values are shown in Table 1. Four different phosphate solutions were used to precipitate the excess silver from the extracts. Two of the phosphate solutions were used with the unfiltered extracts and the remaining two phosphate solutions were used with the filtered extracts. The phosphate solutions were prepared and used as follows:—

(1) 13·6 g KH_2PO_4/100 ml at pH 6·5. Add 1 ml to 25 ml unfiltered extract (Johnson and Ulrich).

(2) 13·6 g KH_2PO_4/100 ml at pH 7·2. Add 1 ml to 25 ml unfiltered extract (modified Johnson and Ulrich).

(3) 4 g KH_2PO_4 plus 1·2 g NaOH/100 ml. The pH of this solution is about 10·5. Add 5 ml to 20 ml filtered extract.

(4) 4 g KH_2PO_4/100 ml at pH 7·2. Add 5 ml to 20 ml filtered extract.

The pH values of the solutions, after the addition of the phosphate solutions to the extracts, are shown in Table 1 as the pH Final values correspondingly numbered with the number of the phosphate solution used to precipitate the excess silver.

From Table 1, it can be seen that the Final pH (1) and Final pH (2) values produced by the addition of phosphate solutions (1) and (2) will not allow complete precipitation of silver, since pH is less than 5·8. Phosphate solution (1) is used in the method of Johnson and Ulrich and phosphate solution (2) is a modified (1) prepared at a higher pH level. Phosphate solutions (3) and (4) are suitable for use but in practice solution (3) is more convenient to use because the removal of the precipitated phosphate is more rapid and efficient from the solution with the higher Final pH level.

Because of the poor keeping quality of aqueous silver sulphate solutions, solid silver sulphate is used in the extraction. A modified extraction procedure was developed in which the sample (0·1—0·3 g), 1 g calcium sulphate and a weight of solid silver sulphate equal to the sample weight are shaken for 15 min with 25 ml water in a 100-ml Erlenmeyer flask. The extracts are filtered through a 9-cm Whatman No. 42 paper. To 20 ml of the clear filtrate in a centrifuge cup, add 5 ml phosphate solution (3). Centrifuge the solution until the liquid is clear of all silver phosphate. An aliquot of the clear solution is then treated according to the method of Johnson and Ulrich.

When the nitrate colour is developed, the sample solutions should be read against blank solutions which have been prepared in the same way as the sample being analysed. If the optical density of the nitrate colour exceeds 0·5, it is advisable to re-analyse the sample, using suitably adjusted smaller aliquots.

TABLE 1

pH Changes of Extracts with the Removal of Excess Silver

EXTRACT COMPOSITION				pH VALUE							
Ag_2SO_4 (g)	$CaSO_4$ (g)	H_2O (ml)	Plant Material (g)	EXTRACT	FINAL* (1)	EXTRACT	FINAL* (2)	EXTRACT	FINAL* (3)	EXTRACT	FINAL* (4)
0·2	1	25	BLANK	6·2	2·7	6·5	3·5	6·5	6·8	6·5	5·7
0·2	1	25	0·2	4·8	5·5	4·8	5·8	4·7	7·4	4·8	6·5
0·1	1	25	BLANK	6·5	5·2	6·6	5·5	6·6	7·2	6·6	6·3
0·1	1	25	0·1	4·9	5·3	4·9	5·5	4·9	7·3	4·9	6·4

* pH of the solution after the addition of the correspondingly numbered phosphate solution to the extract.

REFERENCE

JOHNSON, C. M., and ULRICH, A. (1950).—*Ind. Engng Chem. Analyt. Edn* 22:1526.

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