

1917

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Recommended Citation

Dox, Arthur W. (1917) "The Behavior of Benzidine toward Selenic and Telluric Acids," *Proceedings of the Iowa Academy of Science*, 24(1), 537-538.

Available at: <https://scholarworks.uni.edu/pias/vol24/iss1/75>

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THE BEHAVIOR OF BENZIDINE TOWARD SELENIC AND TELLURIC ACIDS.

ARTHUR W. DOX.

Within quite recent years benzidine (*p*-diaminodiphenyl) has come into use as a precipitant for the sulfate ion. It was first applied as a quantitative reagent for the determination of sulfate by Raschig¹ in 1903. Other investigators subsequently introduced modifications in the original method of Raschig and succeeded in obtaining very satisfactory analyses with this reagent. For example, in the analysis of water samples which contain iron salts, hydroxylamine hydrochloride is added to prevent oxidation of the benzidine. The precipitated benzidine sulfate is collected in the usual way and is either weighed direct or titrated with sodium hydroxide, using phenolphthalein as an indicator. Titration is rendered possible by the very weak basic properties of benzidine. Bruckmiller² states that the benzidine method for sulfates in water compares favorably with the time-honored barium chloride method in point of accuracy, and has the advantage of being more rapid.

The writer undertook to determine whether the corresponding acids of selenium and tellurium, two elements closely analogous to sulfur and occurring in the same group of the periodic system, would react in the same manner, with benzidine.

The benzidine reagent was prepared as follows: Two grams of Merck's benzidine were stirred to a paste with a little water, washed into a 250 cc. volumetric flask, 2.5 cc. concentrated hydrochloric acid added, and the solution made up to the mark. A slight sediment was removed by filtration.

Qualitative tests were first made with this reagent. When added to a solution of sodium sulfate, as was expected, a white granular crystalline precipitate began to form instantly. With a solution of Kahlbaum's sodium selenate the same phenomenon was observed, except that the precipitate was more granular and settled out more readily. However, a solution of Kahlbaum's sodium tellurate gave no precipitate whatever with the benzidine reagent.

¹Raschig, *Z. angew. Chem.*, 1903, 617, 818.

²Bruckmiller, *J. Ind. Eng. Chem.*, 7, 600, 1915.

The two precipitates above mentioned were then prepared in larger quantities, and after careful washing and drying were further identified by analyses for nitrogen.

SUBSTANCE	NITROGEN	
	FOUND	CALCULATED
Benzidine sulfate	9.61 9.53	9.93
Benzidine selenate	8.40 8.34	8.51

In order to determine how nearly quantitative was the precipitation of selenic acid, a stock solution of the sodium selenate was prepared by dissolving one gram of the crystalline substance in 100 cc. water. Portions of 10 cc. each of this solution were used for the determinations given below, which were carried out in the usual way, using both the benzidine and the barium chloride methods. Parallel determinations were made with a similar solution of sodium sulfate.

SODIUM SULFATE

BaSO ₄ FOUND	SO ₄ CALC.	BENZIDINE SULFATE	SO ₄ CALC.
		FOUND	
.1289	.0531	.1552	.0528
.1288	.0531	.1548	.0527

SODIUM SELENATE.

BaSeO ₄ FOUND	SeO ₄ CALC.	BENZIDINE SELENATE	SeO ₄ CALC.
		FOUND	
.0749	.0383	.0829	.0360
.0749	.0383	.0832	.0362
		.0821	.0357
		.0819	.0356

From the above data it will be seen that sulfate determined as benzidine sulfate agrees fairly well with that determined as barium sulfate, as has been claimed by other investigators. However benzidine selenate, prepared here for the first time, is not precipitated as completely as the barium selenate. The average of the four determinations shows that under the conditions of the experiment about 94 per cent of the selenate is precipitated by benzidine, assuming that the precipitation of barium selenate is quantitative. In the presence of dilute hydrochloric acid, tellurates give no precipitate with benzidine.

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