

DETERMINATION OF THE MAIN COMPONENTS OF SEMICONDUCTING COMPOUNDS OF INDIUM WITH ARSENIC AND PHOSPHORUS

Yu. S. Lyalikov and K. E. Kolchina

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Institute of Chemistry, Academy of Sciences of the Moldavian SSR

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A method has been developed for the analysis of InP, InAs, and InP-InAs alloys, for their main components In, As, and P, without preliminary separation.

Ammonium citrate, which also acted as a buffer, was used to eliminate the interfering effects of AsO_4^{3-} and PO_4^{3-} on the determination of In.

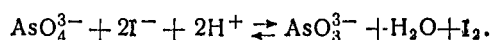
In the present paper we give methods and results for the analysis of binary InP and InAs alloys, and of ternary InP-InAs alloys.

In order to convert arsenic and phosphorus to the pentavalent state, the sample was dissolved in nitric acid, or in a mixture of nitric and hydrochloric acids, in a flask with a reflux condenser. Indium can be determined by the direct complexometric volumetric methods with Xylenol Orange as indicator [1]. However, low results for indium were obtained in the analysis of InP and InAs alloys, because of the formation of indium arsenate and phosphate at the pH used. However, addition of ammonium citrate to the solution before the titration prevented deposition of the above compounds and made the normal titration possible. Moreover, addition of this reagent made it possible to maintain the requisite pH of 3.5 [2].

In the case of InP alloys, the phosphorus was determined gravimetrically by the magnesium method [3] immediately after the indium determination, and on the same solution.

In the case of InAs alloys, AsO_4^{3-} was determined by the volumetric iodometric method [4, 5] after the indium determination, and on the same solution.

In the case of InP-InAs alloys, the As(V) was reduced to As(III) in accordance with the reaction:



and this created conditions suitable for the determination of PO_4^{3-} in the presence of AsO_3^{3-} . Thus the phosphorus was determined by the magnesium method [3] after the determination of arsenic.

METHOD OF ANALYSIS

A 0.4 to 0.5 g sample of InAs, InP, or InP-InAs alloy was dissolved in nitric acid, or in a mixture of nitric and hydrochloric acids. The solution was evaporated to dryness, and the dry residue was dissolved in dilute sulfuric acid, transferred to a 100 ml graduated flask, and made up to the mark. A 5 to 10 ml aliquot of this was taken for analysis, diluted to 15 to 20 ml, and treated with ammonium citrate to give a pH of 3.5. The solution was heated to 50 to 60°C and treated with two drops of the indicator Xylenol Orange, and the indium was titrated with 0.2 M trilon B until the color changed from red-violet to lemon-yellow.

For the determination of PO_4^{3-} in InP alloys, the solution which had been used for indium determination was treated with 10 ml of HCl (1:19) and 100 ml of magnesia mixture (100 g $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$, 125 g NH_4Cl , 150 ml H_2O , 500 ml NH_3 solution), and the solution was neutralized with ammonia and treated with 3 ml in excess. The solution was mixed and allowed to stand for a few hours, and the precipitate was then filtered off, dissolved, reprecipitated and allowed to stand for 4 h. The precipitate was then filtered off on a fine paper, washed with dilute ammonia (1:19), placed in a weighed porcelain crucible, and ignited at 1050 to 1100°C.

Results for the Determination of In, P, and As in Samples of InP, InAs, and InP-InAs

Alloy analyzed	Theo. content, %			Mean of several determinations, %		
	In	P	As	In	P	As
InP	78.8	21.2	—	78.6±0.4	21.7±0.8	—
In As	60.5	—	39.5	60.8±1.0	—	39.6±1.0
InP—InAs . . .	—	—	—	63.8±0.6	2.4±0.3	34.7±0.9
InP—InAs . . .	—	—	—	68.7±0.8	11.2±0.5	19.5±1.0

For the determination of AsO_4^{3-} in InAs alloys, the solution which had been used for indium determination was treated in a flask with 20 to 25 ml of HCl, cooled, and treated twice with 0.5 g of NaHCO_3 to eliminate interference by oxygen. It was then treated with 1 g of KI, and the flask was covered by a test tube and allowed to stand in the dark for not less than 5 min. The iodine liberated was titrated with 0.1 N thiosulfate solution until the blue color with starch disappeared.

For the analysis of InP-InAs alloys, the solution which had been used for indium determination was first analyzed for AsO_4^{3-} and then for PO_4^{3-} as described above.

The table shows some results obtained.

LITERATURE CITED

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