Title

Studies on the Fluorometric Analysis. (VI) : Fluorometric Determination of Gallium, Indium and Beryllium by Successive Extraction

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ABSTRACTS
(Ishibashi Laboratory)

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Optimum conditions for the fluorometric determination of aluminum by the use of Pontachrome Blue Black R have been investigated. For aluminum solutions of pH 4.8 and aluminum content 0.2-1.0 g, 1-12 g, and 12-18 g, 1.0 ml, 1.5 ml, and 2.0 ml, respectively, of 1% aqueous solution of Pontachrome Blue Black R were added. Each solution was heated for 10 minutes on a water bath, cooled, made up to 50 ml, and measured its intensity of fluorescence. The interfering elements in the determination of aluminum by this method are \( \text{Fe}^{3+} \), \( \text{Ga} \), \( \text{Co} \), and vanadic acid. Also, the presence of a large amount of \( \text{Cu} \), \( \text{Ti} \), and \( \text{Ni} \) has a disturbing influence.

**Studies on the Fluorometric Analysis. (V)**

**Determination of Gallium with 8-Hydroxyquinaldine**

Masayoshi Ishibashi, Tsunenobu Shigematsu and Yasuharu Nishikawa
(Ishibashi Laboratory)


8-Hydroxyquinaldine reacts with gallium ion in weak acidic solution and its chloroform extracts show a distinct green fluorescence in ultraviolet light. Using this reaction, the authors established the fluorometric method for trace amounts of gallium as follows:

Samples containing 0-30 \( \mu \)g of gallium in a volume of approximately 40 ml were treated with 1 ml of 1%±8-hydroxyquinaldine-1N-acetic acid, and was extracted three times with 10 ml portions of chloroform. The extracts were diluted to 50 ml with chloroform and the fluorescence intensity was measured.

A few elements such as cupric cupper, tartaric acid, and large amounts of indium and thallium are interfered. Ferric ion and Vanadate ion are also interfered, however, these ions are easily reduced by adding 1-2 ml of 1% hydroxylamine hydrochloride solution and gallium quantified without interference.

**Studies on the Fluorometric Analysis. (VI)**

**Fluorometric Determination of Gallium, Indium and Beryllium by Successive Extraction**

Masayoshi Ishibashi, Tsunenobu Shigematsu and Yasuharu Nishikawa

(111)
Gallium, indium and beryllium-8-hydroxyquinaldate are easily extracted with chloroform, and these complexes were completely extracted from the solution of following pH range:
Ga-complex: pH 3.9-5.0, In-complex: pH 5.5, Be-complex: pH 7.6-8.6.
Accordingly, the authors carried out a successive extraction of these metalloquinaldate complexes with chloroform at optimum extraction pH (i.e. Ga: 3.9, In: 5.5 and Be: 8.2) and by this method, 2-10 μg Ga, 25-100 μg In and 2-5 μg Be/50 ml are determined respectively.

The Use of Radioactive Element. (II)
Decomposition of 1,4-Dibenzoylthiosemicarbazide and 1,4-Dibenzoylsemicarbazide with Alkali
Risaburo Nakai, Michiyasu Sugii and Hideo Nakao
(Nakai Laboratory)
Pharmaceutical Bulletin, 5, 576 (1957)

1,4-Dibenzoylthiosemicarbazide (II) or 1,4-dibenzoylsemicarbazide (V) is converted by the action of alkali into 3-phenyl-5-mercapto-1,2,4-triazole (IV) or 3-phenyl-5-hydroxy-1,2,4-triazole (VII), benzoic acid and ammonia. There are two possible routes in this reaction, since IV is prepared from both 1-benzoyl-thiosemicarbazide (A) and 4-benzoylthiosemicarbazide (B) and VII is prepared from both 1-benzoylsemicarbazide (A') and 4-benzoylsemicarbazide (B'). These monobenzoyl compounds are presumed as intermediate products of the reaction. The reaction routes were studied by using C\textsuperscript{14}-tracer technique. Labeled compounds II and V were synthesized from benzohydrazide (carbonyl-C\textsuperscript{14}) and were treated with 10% NaOH and 20% NaOH, respectively. The specific radioactivities (μC/m.mole) of the products measured at each step are shown under the formulae in Chart 1.

Chart 1

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\begin{align*}
\text{(I)} & \quad \text{C}_6\text{H}_5\text{CONH}\cdot\text{NHCONHCOC}_6\text{H}_5 \rightarrow \text{C}_6\text{H}_5\text{COOH} + \text{C}_6\text{H}_5\cdot\text{CONH} \cdot \text{NHCONHCOC}_6\text{H}_5 \quad 4.05 \pm 0.04 \\
\text{(II)} & \quad \text{G}_6\text{H}_6\cdot\text{CONH} \cdot \text{NHCSNHCOC}_6\text{H}_5 \rightarrow \text{C}_6\text{H}_5\text{COOH} + \text{C}_6\text{H}_5\cdot\text{CONH} \cdot \text{NHCONHCOC}_6\text{H}_5 \quad 4.08 \pm 0.11 \\
\text{(III)} & \quad \text{C}_6\text{H}_5\cdot\text{CONH} \cdot \text{NHCONHCOC}_6\text{H}_5 \rightarrow \text{C}_6\text{H}_5\text{COOH} + \text{C}_6\text{H}_5\cdot\text{CONH} \cdot \text{NHCONHCOC}_6\text{H}_5 \quad 4.09 \pm 0.08 \\
\text{(IV)} & \quad \text{N}_2 \text{H}_4 \cdot \text{SH} \quad 4.08 \pm 0.12 \\
\text{(V)} & \quad \text{N}_2 \text{H}_4 \cdot \text{SH} \quad 4.08 \pm 0.08 \\
\text{(VI)} & \quad \text{N}_2 \text{H}_4 \cdot \text{SH} \quad 1.76 \pm 0.04 \\
\text{(VII)} & \quad \text{N}_2 \text{H}_4 \cdot \text{SH} \quad 4.06 \pm 0.07
\end{align*}
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