MinistryofHealthoftheRussianFederation Volgograd State Medical University

DepartmentofPharmaceuticalandToxicological Chemistry

GENERAL PHARMACEUTICAL CHEMISTRY

CHEMICALMETHODSOFPHARMACOPOEIAL ANALYSIS - IDENTIFICATION OF DRUGS OF ORGANIC NATURE (IDENTIFICATION OF FUNCTIONAL GROUPS) (Identification of carboxyl, ester and amide groups)

Lesson6

V term

Volgograd,2022

IDENTIFICATION OF CARBOXYL, ESTER AND AMIDE GROUPS

Identification of the carboxyl group

R-COOH

1. Reactions with heavy metal salts.

The medicinal substance reacts only in ionized form, so it is first converted to sodium (or ammonium) salt. The reaction of the medium should be close to neutral, so the alkali is taken in an equivalent amount (without excess). In an alkaline medium, the reagents are precipitated in the form of hydroxides, they do not react with the medicinal substance.

Reactions of this group are insensitive, so it is necessary to prepare a concentrated solution of the medicinal substance (usually 2-5%).



Mostoften, heavy metals alt shave the following colors:

- \checkmark silversaltsarewhite,
- \checkmark mercurysaltsare gray,
- ✓ iron(III)saltsarepinkish−yellow,
- ✓ copper(II)saltsareblueorblue,
- ✓ cobaltsaltsarelilacorpink.

2. Esterification reactions

Esterification is carried out in the presence of dehydrating substances (concentrated sulfuric acid). Very widely used, for example, the reaction of the formation of ethyl acetate, which has a peculiar fruity smell.



Identification of the estergroup



1. Hydrolysis.

Hydrolysis is carried out in anacidicoralkalinemedium.



2. Reaction of formation of hydroxamic acids (hydroxamictest).

Hydroxamic acids with iron (III) and copper (II) salts in an acidic medium form colored complex salts – iron (III) hydroxamates (cherry, red-brown or red-purple) and copper (II) hydroxamates (green or blue-green).



Identification of amide and sulfamide groups



1. Decomposition reactions of amides occur when heated in solutions of caustic alkalis with the formation of ammonia or alkylamines having a characteristicsmell:



These chemical reactions are used to test the identity of salts of primary ammonium bases, amides of aromatic and heterocyclic acids, derivatives of urethanes.

Identification of the secondary amino group in the composition of imide, sulfamide, hydrazide functional groups and in some heterocyclic compounds Complexation reactions.

Reagents are salts of silver, copper (II), iron (III), cobalt (II), mercury (II), nickel, lead and other metals.

The effect of the reaction is the formation of compounds that are difficult to dissolve in water. The color is determined by the color of the medicinal substance and reagent, as well as the structure of the resulting complex compound. Precipitation may have a characteristic crystal shape.

Thus, barbiturates are characterized by the following chemistry of complex formation:



$R-CH=CH-R+2KMnO_4+4H_2O\rightarrow 3R-CH-CH-R+2MnO_2+2KOH$ $\begin{vmatrix} & \\ & \\ & \\ & \\ & \\ & OH & OH \end{vmatrix}$

Detecting multiple bonds

1. Bromination. 1 ml of the test substance solution is placed in a dry test tube and a brominesolution is added drop bydrop,with light shaking. The disappearance of the yellow color is observed:



2. Test with potassium permanganate (Wagner reaction). In a slightly alkaline medium, under the action of potassium permanganate, the substance is oxidized with the rupture of a multiple bond.