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APPLICATION OF INFRARED SPECTROSCOPY AND X-RAY POWDER DIFFRACTOMETRY FOR ASSESSMENT OF THE QUALITATIVE COMPOSITION OF COMPONENTS IN A PHARMACEUTICAL FORMULATION

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Abstract. A qualitative assessment of a new four-component pharmaceutical composition has been carried out using the methods of infrared spectroscopy and X-ray powder diffractometry. Qualitative characteristics for the identification of mixture components by absorption bands in infrared spectra and characteristic peaks by positions on the scattering angle 2θ scale in diffractograms were determined. It was experimentally confirmed that the quantitative content of benzocaine and procaine hydrochloride in the mixture without diclofenac sodium decreased by two times compared to their content in the mixture with it. Original infrared spectra and X-ray diffractograms of the new pharmaceutical composition with diclofenac sodium, which can be used for its identification, are presented.

Keywords: active pharmaceutical ingredient (API), quality composition, API mixture, infrared spectroscopy, X-ray powder diffraction.

1. Introduction

The development of new medicinal products based on known or new active pharmaceutical ingredients always involves developing and applying various analytical quality control methods for their unambiguous identification. The use of such well-known methods as infrared spectroscopy and X-ray powder diffraction is a powerful and indicative tool for pharmaceutical analysis.[1]

The study of compounds by the method of absorption spectrophotometry in the infrared region is widely used to identify various classes of substances, determining the structure of individual molecules[2-8] and the composition of molecular mixtures in industrial and scientific laboratories.[9] Moreover, IR-Fourier spectroscopy makes it possible to understand better the kinetics, mechanisms, and pathways of chemical reactions. It is used to check the conformity of raw materials, intermediate and final products, including the determination quality of API for medicinal products.[10]

The X-ray powder diffraction (XRD) method is used at various stages of quality control in the production of medicinal products. It allows to evaluate the mechanical and chemical activation of solid substances, investigate the nature of composites with altered reactivity,[11,12] polymorphism, and record changes in the biological activity of substances and medicinal products.[13] At the same time, XRD as a method of pharmaco-technological research was introduced to assess the qualitative and quantitative phase composition of tested substance samples.[10,14] This method is included in the world's leading pharmacopeias, such as The United State Pharmacopoeia USP 42, European Pharmacopoeia 10, and Japanese Pharmacopoeia JP XVIII. However, in Ukraine, the XRD method is still rarely used in the identification of active substances. This test method found its use for API research in various works of an analytical nature.[1,15-17] X-ray powder diffraction is a valuable method for pharmaceutical analysis, including the control of counterfeit drugs.[18-20]

The advantages of the methods mentioned above are the ease of preparing samples for research and the speed of obtaining results.[21]

Therefore, the purpose of this work is to conduct a qualitative analysis of a new pharmaceutical composition using the methods of infrared spectroscopy and X-ray powder diffraction for the possibility of its identification in the mixture components.

2. Experimental

Active pharmaceutical ingredients (APIs): procaine hydrochloride (novocaine hydrochloride), racemic menthol, and benzocaine (>99%) were purchased from Istok-Plus LLC (Ukraine). Diclofenac sodium (99.5 %) was

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purchased from Amoli Organics Pvt. Ltd. (India). Infrared spectra were recorded on a Spectrum Two IR-Fourier spectrometer (PerkinElmer, USA). Registration of IR spectra was carried out with the help of a universal attachment of impaired total internal reflection (UATR) with a diamond crystal in the region from 4000 to 400 cm^{-1}. Diffractograms were recorded using an AERIS Research X-ray diffractometer (Malvern PANalytical, The Netherlands). X-ray diffractograms were recorded with CuKα radiation at 50 kV and 15 mA in the range of 2θ angles from 7 to 70. The components of the pharmaceutical composition were studied in the powder state.

3. Results and Discussion

For the qualitative assessment of components in a new pharmaceutical composition (API powder mixture), which consists of procaine hydrochloride, menthol, benzocaine, and diclofenac sodium, we chose methods of absorption spectrophotometry in the infrared region and X-ray diffraction of powder, which allows for quick and qualitative identification of the tested sample composition. The research subjects were samples of menthol, procaine hydrochloride, benzocaine and diclofenac sodium APIs and their mixtures.

3.1. Qualitative Determination of Composition Components by Absorption Spectrophotometry in the Infrared Region

Identification tested samples of procaine hydrochloride, benzocaine, and diclofenac sodium in the corresponding pharmacopoeial articles performed by IR spectroscopy using a comparison with the corresponding spectra of pharmacopoeial standard samples.[22] To unambiguously identify all APIs of the tested pharmaceutical composition by the method of absorption spectrophotometry in the infrared region, IR spectra of the following samples were recorded: individual powder substances of menthol, procaine hydrochloride, benzocaine, diclofenac sodium; powder mixture of APIs without diclofenac (menthol + procaine hydrochloride + benzocaine) and powder mixture of APIs with diclofenac sodium (menthol + procaine hydrochloride + benzocaine + diclofenac sodium).

Comparison of absorption bands in IR spectra and identification by these parameters for individual substances of procaine hydrochloride, menthol, benzocaine, and diclofenac sodium (Figs. 1-4) was carried out using the known IR spectra of the indicated APIs obtained from the *SciFinder-n* database *and PerkinElmer Spectrum 10 Spectroscopy Software*.

As a result, it was established that the position of the absorption bands and their relative values in the investigated IR region for the corresponding tested API and its standard spectrum from the database are entirely consistent. After identifying individual APIs by the characteristic values of absorption bands, the IR spectra of the following mixtures were recorded: **1** – menthol + procaine hydrochloride + benzocaine (Fig. 2), **2** – menthol + procaine hydrochloride + benzocaine + sodium diclofenac (Fig. 3).

The analysis of the IR spectrum of mixture **1** (Fig. 2) showed preservation of the main absorption bands characteristic of each API (procaine hydrochloride, menthol, and benzocaine), for which valence vibrations of NH_2 (procaine and benzocaine) and OH (menthol) functional groups were recorded in the region of 3419-3206 cm^{-1}. Absorption bands at 2955-2869 cm^{-1} region are characteristic of asymmetric and symmetric valence vibrations of -CH- aliphatic CH_2 groups in procaine molecules and asymmetric valence vibrations of –CH– of the methyl group in menthol. Also, absorption bands corresponding to the tertiary ammonium salt of procaine hydrochloride are recorded in the range of 2585-2495 cm^{-1}. Valence vibrations of ester carbonyl groups of procaine hydrochloride and benzocaine were observed as absorption bands of fragments in the region of 1689-1616 cm^{-1} at 1678 cm^{-1} and 1689 cm^{-1}, respectively. In the IR spectrum of mixture **1**, bands of valence absorptions -C=C- of the aromatic ring are recorded at 1635 cm^{-1}. Asymmetric vibrations of –CH– in the CH_3 group and deformation linear vibrations of –C-C– and symmetric deformation vibrations of –CH– in the methyl group of menthol molecule were recorded as absorption bands at 1463 cm^{-1} and 1443 cm^{-1}, respectively. An absorption band characteristic of the valence vibrations of the aliphatic and aromatic –C–N– bond of procaine hydrochloride and benzocaine was detected at 1267 cm^{-1}. In the case of ester fragments of procaine hydrochloride and benzocaine –C-O-C–asymmetric valence vibrations are presented in the range of 1272-1170 cm^{-1}. In turn, the characteristic absorption band of –C-OH valence vibrations for menthol in mixture **1** is observed at 1045 cm^{-1}. Moreover, the absorption bands for the deformation vibrations of the =CH– aromatic ring and alkyl fragments of procaine hydrochloride and benzocaine are reordered in the range of 846-770 cm^{-1}.

The analysis of the IR spectrum of mixture **2** (Fig. 3) showed the appearance of other, unlike those described above, bands of valence vibrations of the following groups of atoms, which correspond to the diclofenac sodium molecule. In particular, the secondary amino group –NH– is observed at 3386 cm^{-1}, while asymmetric and symmetric vibrations of the sodium salt of the carboxyl group $-COO^-$ are reordered at 1573 cm^{-1} and 1397 cm^{-1}, respectively. In

the case of -C-Cl fragment vibrations are presented at a 754 cm^{-1}. The absorption bands of aryl =CH and methylene group are kept in the corresponding areas of 3419-3206 cm^{-1} and 2955-2869 cm^{-1}.

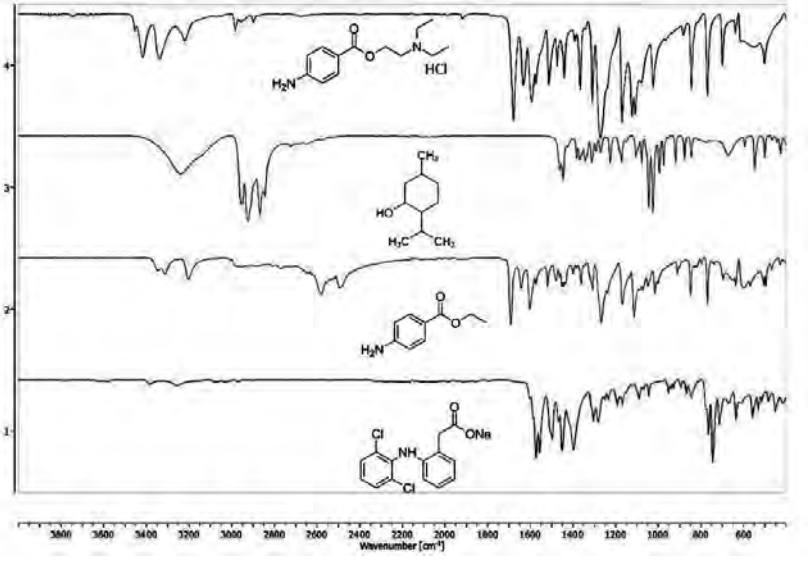

Fig. 1. FT-IR (UATR) spectra of diclofenac sodium (1), benzocaine (2), menthol (3), and procaine hydrochloride (4)

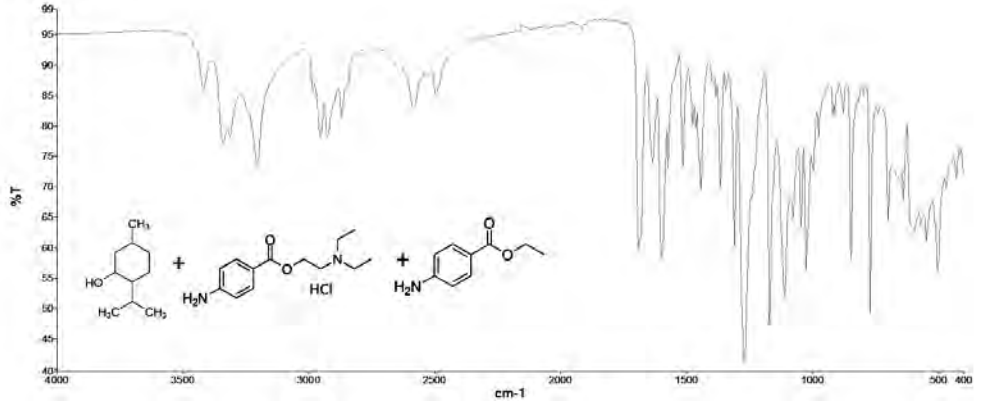

Fig. 2. FT-IR (UATR) spectrum of mixture **1** (menthol + procaine hydrochloride + benzocaine)

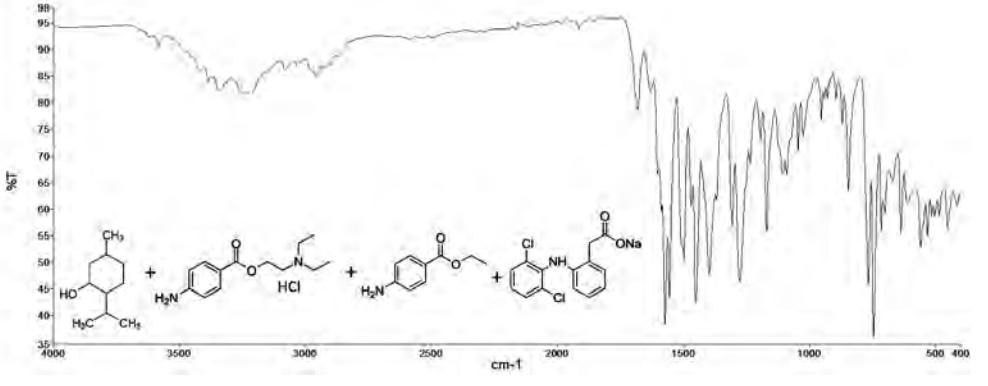

Fig. 3. FT-IR (UATR) spectrum of mixture **2** (menthol + procaine hydrochloride + benzocaine + diclofenac sodium)

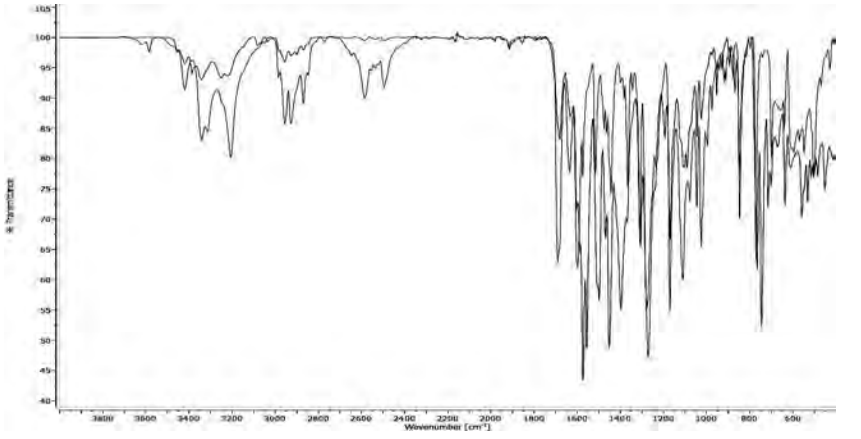

Fig. 4. Overlay of FT-IR (UATR) spectra of mixtures **1** (green color) and **2** (purple color)

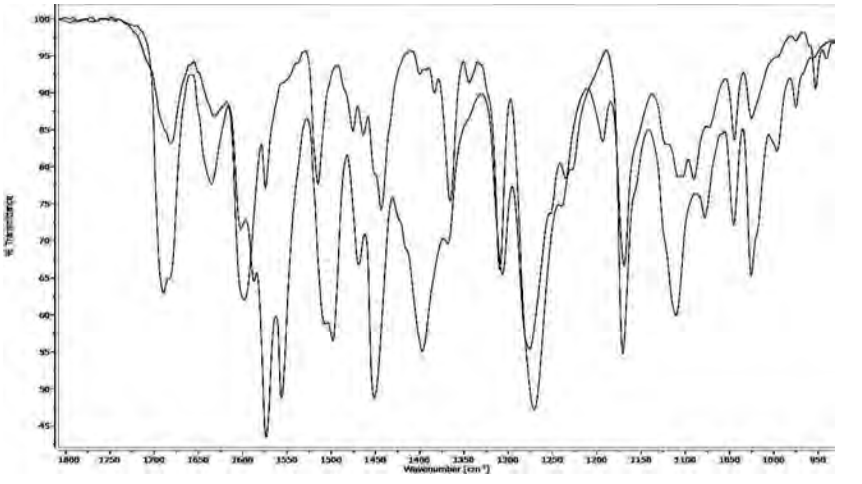

Fig. 5. Overlay of FT-IR (UATR) spectra of mixtures **1** (green color) and **2** (violet color) in the regions of 1659-1616 and 1739-1655 cm^{-1}

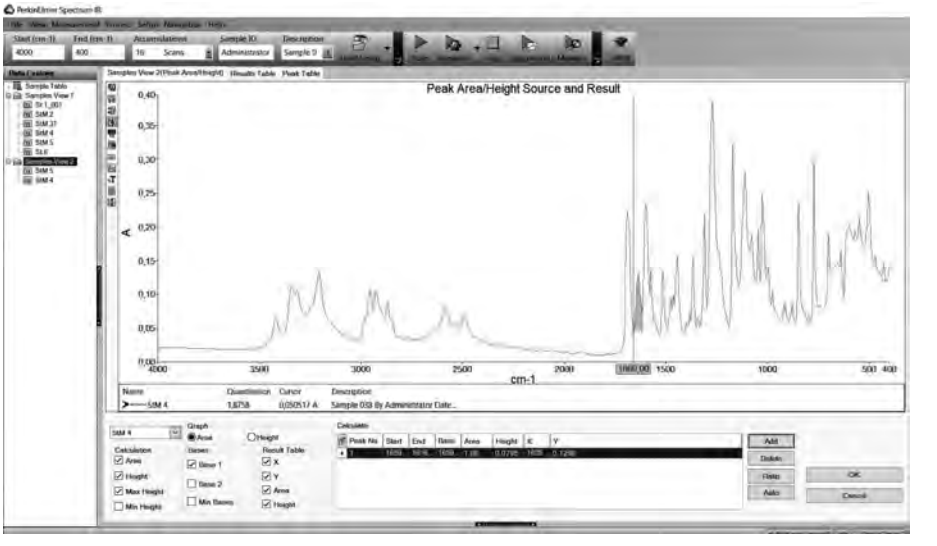

Fig. 6. Measurement of the area of the absorption band in mixture **1** in the region of 1659-1616 cm^{-1} using *Perkin–Elmer Spectrum 10 Spectroscopy Software*

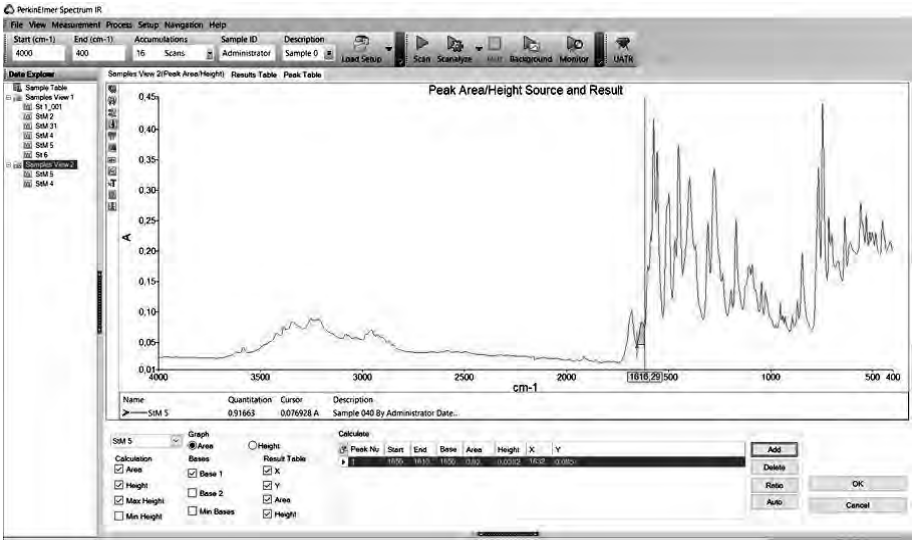

Fig. 7. Measurement of the area of the absorption band in mixture **2** in the region of 1659-1616 cm^{-1} using *Perkin–Elmer Spectrum 10 Spectroscopy Software*

Comparison of the FT-IR (UATR) spectra of mixtures 1 and 2 (Figs. 4 and 5) made it possible to quantitatively assess the decrease in procaine hydrochloride and benzocaine content in mixture **2**. The decrease was identified according to the most optimal and visually convenient indicator by measuring the areas of absorption bands of carbonyl groups in the ranges of 1659- 1616 cm^{-1} (Figs. 6 and 7) and 1739-1655 cm^{-1}.

As a result of measuring the areas, the following data were obtained. In the section of 1659-1616 cm^{-1}, this value was 1.88 for mixture **1** and 0.92 for mixture **2**. In the absorption region of 1739-1655 cm^{-1}, the index for mixture **1** was 6.08, and for mixture **2** – 3.11. Thus, using a comparison of the areas of the bands of valence vibrations of the carbonyl groups of benzocaine and procaine hydrochloride, it was experimentally confirmed that the quantitative content of these APIs decreased by two times in mixture **2** compared to their content in mixture **1**.

3.2. Qualitative Determination of Composition Components by X-Ray Diffraction Method

Qualitative API identification of the obtained spray was carried out by the XRD method.[10] Samples of the tested APIs were prepared in a similar way as in the case of studies using FT-IR: individual powder substances of procaine hydrochloride, menthol, benzocaine, diclofenac sodium; powder mixture of AFI without diclofenac sodium (menthol + procaine hydrochloride + benzocaine) and powder mixture of AFI with diclofenac sodium (menthol + procaine hydrochloride + benzocaine + diclofenac sodium).

At the first stage, the recorded diffraction patterns (Fig. 8, samples 1, 2, 3, 6) of each of the four APIs were compared with the diffraction patterns known in the literature and *Malvern Panalytical's software*. In the second stage, the qualitative content of the components in the mixtures was determined without diclofenac sodium (Fig. 8, sample 4) and with diclofenac sodium (Fig. 8, sample 5).

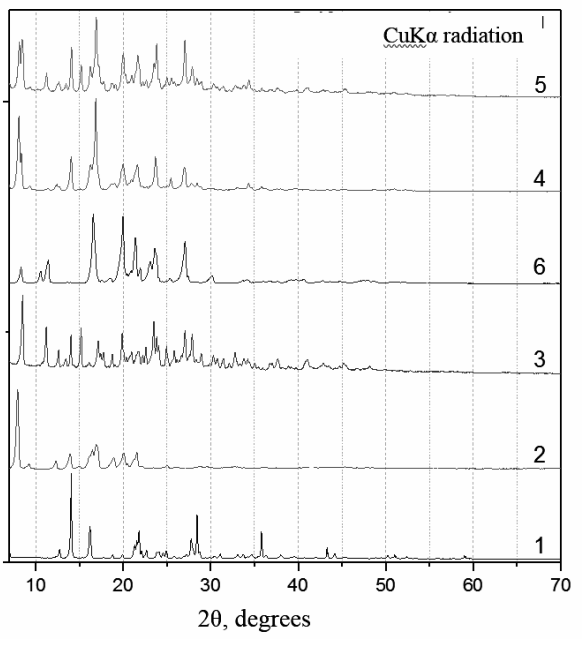

Fig. 8. X-ray diffractograms of procaine hydrochloride (1), menthol (2), diclofenac sodium (3), benzocaine (6); powder mixture of API without diclofenac sodium (4) and powder mixture of API with diclofenac sodium (5)

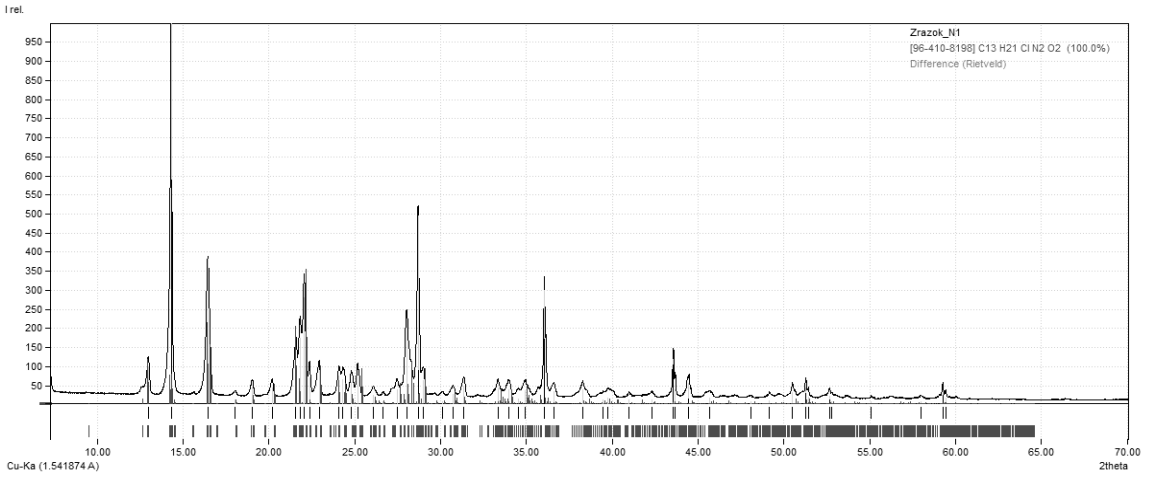

Fig. 9. Superimposition of the experimental diffractogram of sample 1 and the theoretical one for procaine according to the COD database[24] (COD ID 4108197)

The results of the first stage showed the following. Overlaying the experimental diffractogram of sample 1 and the theoretical one for procaine in the *PowderCell* program[23] gave a picture of the coincidence of peaks between the theoretical diffractogram of procaine hydrochloride and the experimental diffractogram for sample **1**. Thus, this sample **1** contains procaine (Fig. 9).

Qualitative identification of menthol (sample 2) was made based on a comparison with the diffractogram for menthol given in the article.[25] The superimposition of this diffractogram with the experimental diffractogram for sample 2 gave a picture of the coincidence of peaks between the diffractogram from article[25] and the experimentally obtained diffractogram. Thus, it was concluded that sample 2 corresponds to menthol (Fig. 10).

The qualitative assessment of sample 3 as diclofenac sodium was carried out based on a comparison of the recorded diffractogram with the diffractogram of diclofenac sodium presented in article[26] (Fig. 11). As a result, the coincidence of the peaks between the diffractogram in the mentioned article and the experimentally recorded diffractogram for sample 3 was established, which is evidence of the content of diclofenac sodium in the tested sample 3.

Similarly, identification was carried out for sample 6 (benzocaine). In the article,[27] the authors recorded three polymorphic forms of benzocaine using powder XRD research. Overlaying the diffractograms of the three forms of the crystal structure of benzocaine with the experimental diffractogram for sample 6 revealed a coincidence of peaks between the diffractograms of forms II and III of benzocaine from the article[22] and the recorded diffractogram for sample 6. This result indicates that sample 6 contains benzocaine in a mixture of two forms of the crystal structure (Fig. 12).

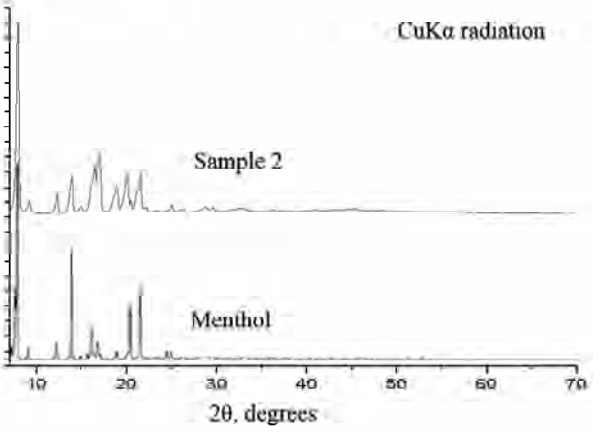

Fig. 10. Comparison of diffractograms of menthol from[25] and sample 2

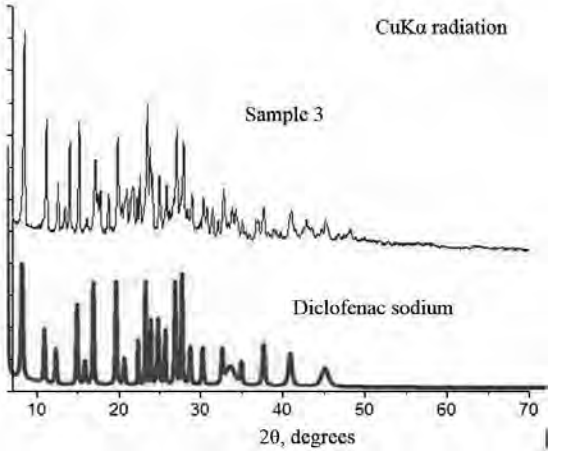

Fig. 11. Comparison of diffractograms of diclofenac sodium from[26] and sample 3

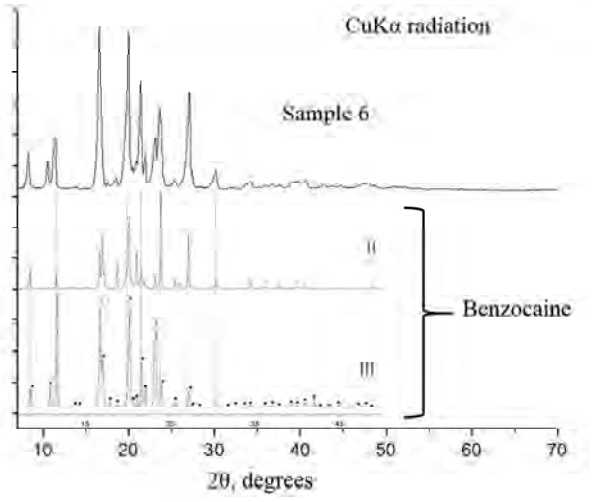

Fig. 12. Comparison of diffractograms of two forms of the crystal structure of benzocaine from[27] and sample 6

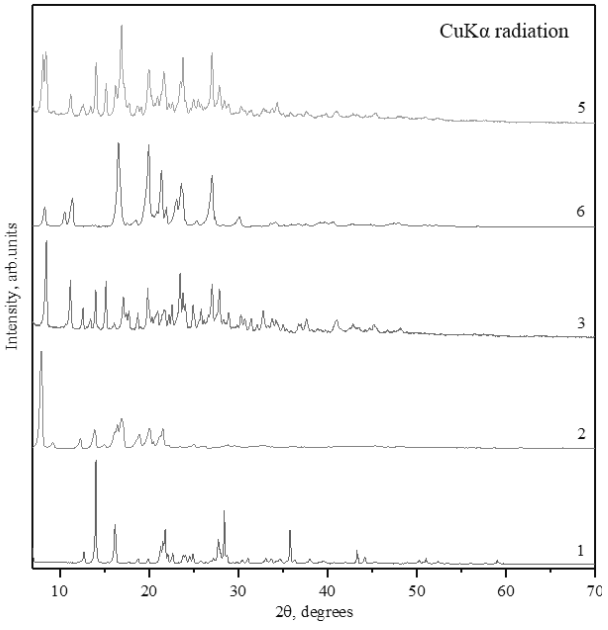

Fig. 13. X-ray diffractograms of procaine hydrochloride (1), menthol (2), diclofenac sodium (3), benzocaine (6), and a powder mixture of API with diclofenac sodium (5)

Determination of the qualitative content of components in mixtures without diclofenac sodium (Fig. 8, sample 4) and with diclofenac sodium (Fig. 8, sample 5) showed the following. The diffractogram of sample 4 (menthol + procaine + benzocaine) shows the peaks observed in the diffractograms of samples 1, 2, and 6 (by positions on the scattering angle 2θ scale). Therefore, the composition of the mixture in sample 4 includes procaine, menthol, and benzocaine. The diffractogram of sample 5 (menthol + procaine hydrochloride + benzocaine + diclofenac sodium) has characteristic peaks at the positions on the scattering angle 2θ scale, which are fixed on the diffractograms of samples 1, 2, 3, and 6 (at the positions on the scattering angle 2θ scale) (Fig. 13). Thus, it can be stated that the composition of sample 5 includes procaine hydrochloride, menthol, benzocaine, and diclofenac sodium.

4. Conclusions

The possibility of using infrared spectroscopy and X-ray powder diffraction is shown for a quick qualitative composition assessment of the new pharmaceutical composition, which consists of four active substances: procaine hydrochloride, menthol, benzocaine, and diclofenac sodium. The obtained IR spectra and diffractograms can be used to determine the identity of the pharmaceutical composition by superimposition and visual comparison with obtained in the future the mixture samples. Sections of valence vibrations bands of diclofenac sodium, procaine hydrochloride, and benzocaine were determined for possible quantitative assessment of the content in the mixture.

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ЗАСТОСУВАННЯ ІНФРАЧЕРВОНОЇ СПЕКТРОСКОПІЇ ТА РЕНТГЕНІВСЬКОЇ ПОРОШКОВОЇ ДИФРАКТОМЕТРІЇ ДЛЯ ОЦІНКИ ЯКІСНОГО СКЛАДУ КОМПОНЕНТІВ ФАРМАЦЕВТИЧНОЇ КОМПОЗИЦІЇ

Анотація. *Проведено якісну оцінку нової фармацевтичної композиції з чотирьох компонентів за допомогою методів інфрачервоної спектроскопії та рентгенівської порошкової дифрактометрії. Визначено якісні характеристики для проведення ідентифікації компонентів у суміші за смугами поглинання в інфрачервоних спектрах і характерними піками за положеннями на шкалі градусів 2θ у дифрактограмах. Експериментально підтверджено зменшення кількісного вмісту бензокаїну та прокаїну гідрохлориду в суміші без диклофенаку натрію у 2 рази порівняно з їхнім вмістом у суміші з ним. Представлено оригінальні інфрачервоні спектри та рентгенівські дифрактограми запропонованої фармацевтичної композиції з диклофенаком натрію, за якими можна проводити її ідентифікацію.*

Ключові слова: *активний фармацевтичний інгредієнт (АФІ), якісний склад, суміш АФІ, інфрачервона спектроскопія, рентгенівська дифракція порошку.*