Erratum: "HPLC-UV Method Development and Validation for Vitamin D₃ (Cholecalciferol) Quantitation in Drugs and Dietary Supplements"

Igor E. Shohin, Evgeniya A. Malashenko, Yuri V. Medvedev, Maria N. Bogachuk, Stanislav A. Kulakov, Maria A. Paleeva


The data on the objects of study of dietary supplements for food "Detrimax Active" and "Detrimax Baby" were removed from the article due to a technical error, namely, the wrong overestimation of the deviation of the actual amount of vitamin D₃ in the article from the manufacturer declared in the indicated samples of dietary supplements to food.

On page 89 in the section "Objects of Research", mentions of dietary supplements to food "Detrimax Active" and "Detrimax Baby" were removed.

**Instead:** For the assay of cholecalciferol with developed method in real samples, the following vitamin drug products: "Aquadetrim", aqueous solution 10 ml by JSC "Medana Pharma" (valid up to 04.2023, batch № 050420); "Aquadetrim", water soluble tablets by JSC "Akrikhin" (valid up to 04.2022, batch № 170420). Vitamin dietary supplements were also investigated: "Ultra-D", chewable tablets by "Pharma Oy" (valid up to 05.12.2022, batch № 1913870002); "Detrimax Baby", 30 ml by "Curtis Heath Caps Sp.z.o.o." (valid up to 11.2022, batch № 1912004); "Detrimax Active", 30 ml by "Curtis Health Caps Sp.z.o.o." (valid up to 11.2022, batch № 1912041); "Detrimax 1000 IU", tablets by "Eagle Nutritional Inc" (valid up to 02.2022, batch № WJ141); "Detrimax 2000 ME", tablets by "Grocam JBL Sp.z.o.o." (valid up to 11.02.2023, batch № 260220).

**Corrected to:** For the assay of cholecalciferol with developed method in real samples, the following vitamin drug products: "Aquadetrim", aqueous solution 10 ml by JSC "Medana Pharma" (valid up to 04.2023, batch № 050420); "Aquadetrim", water soluble tablets by JSC "Akrikhin" (valid up to 04.2022, batch № 170420). Vitamin dietary supplements were also investigated: "Ultra-D", chewable tablets by "Pharma Oy" (valid up to 05.12.2022, batch № 1913870002); "Detrimax 1000 IU", tablets by "Eagle Nutritional Inc" (valid up to 02.2022, batch № WJ141); "Detrimax 2000 ME", tablets by "Grocam JBL Sp.z.o.o." (valid up to 11.02.2023, batch № 260220).

On page 97 in the section "Intralaboratory precision", mentions of dietary supplements for food "Detrimax Active" and "Detrimax Baby" were removed.

**Instead:** The developed and validated method was used for the analysis of the following dosage forms (drug products and biologically active dietary supplements): Drug products:
1. "Aquadetrim", aqueous solution 10 ml by JSC "Medana Pharma" (valid up to 04.2023, batch № 050420).
2. "Aquadetrim", water soluble tablets by JSC "Akrikhin" (valid up to 04.2022, batch № 170420).
Biologically active dietary supplements:
1. "Ultra-D", chewable tablets "Pharma Oy" (valid up to 05.12.2022, batch № 1913870002).
4. "Detrimax 1000 IU", tablets by "Eagle Nutritional Inc" (valid up to 02.2022, batch № WJ141).

All samples were tested within one analytical cycle which allowed to reduce intralaboratory variability of the investigation results. System suitability met the normal values. Chromatograms of both solid and liquid dosage forms did not show peaks interfering with the analysis. Therefore the test results may be considered significant within the established validation characteristics.

The analysis results were summarized in table 8, standardized by parameter contents μg/g for solid dosage forms and μg/ml for liquid dosage forms. The analysis for liquid dosage forms was performed as corrected by density:
- "Aquadetrim", aqueous solution – 1 g/ml.
- "Detrimax Baby" – 0.9437 g/ml.
- "Detrimax Active" – 0.9447 g/ml.

The method error was calculated by equation:

\[ \Delta = 1.96 \cdot \text{СКО}, \]

where MSD – mean square deviation of intralaboratory precision (MSD for liquid forms was 2.9 %, MSD for dry forms was 5.8 %).

The method error for liquid forms (aqueous solutions) was ±5.68 %, for dry dosage forms ±11.37 %, respectively.
Table 8. Results of quantitative determination of vitamin D₃ (cholecalciferol) in dosage forms (drugs and dietary supplements)

<table>
<thead>
<tr>
<th>Drugname</th>
<th>Vitamin D₃ content in the drug (declared)</th>
<th>Vitamin D₃ content in 1 g (1 ml) of the drug (declared)</th>
<th>Vitamin D₃ content in 1 g (1 ml) of the drug (found)</th>
<th>Deviation (found/declared), %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ultra-D 25 μg/tablet (tablet weight 425 mg)</td>
<td>58.8 μg/g</td>
<td>52.6 μg/g</td>
<td>–10.5</td>
<td></td>
</tr>
<tr>
<td>Detrimax Baby 5 μg/1 drop (200 IU/drop)</td>
<td>87.7 μg/ml</td>
<td>87.7 μg/ml</td>
<td>–41.5</td>
<td></td>
</tr>
<tr>
<td>Detrimax Active 12.5 μg/1 drop</td>
<td>250.3 μg/ml</td>
<td>250.3 μg/ml</td>
<td>–33.3</td>
<td></td>
</tr>
<tr>
<td>Detrimax 1000 IU 25 μg/tablet</td>
<td>106.1 μg/g</td>
<td>106.1 μg/g</td>
<td>–2.4</td>
<td></td>
</tr>
<tr>
<td>Detrimax 2000 IU 50 μg/tablet</td>
<td>202.4 μg/g</td>
<td>202.4 μg/g</td>
<td>–2.8</td>
<td></td>
</tr>
<tr>
<td>&quot;Aquadetrim&quot; aqueous solution</td>
<td>370 μg/ml</td>
<td>370 μg/ml</td>
<td>–1.3</td>
<td></td>
</tr>
<tr>
<td>&quot;Akvadetrim&quot; water-soluble tablets</td>
<td>155 μg/g</td>
<td>155 μg/g</td>
<td>–0.6</td>
<td></td>
</tr>
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Corrected to: The developed and validated method was used for the analysis of the following dosage forms (drug products and biologically active dietary supplements): Drug products:
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Biologically active dietary supplements:
1. "Ultra-D" chewable tablets "Pharmia Oy" (valid up to 05.12.2022, batch № 1913870002).
2. "Detrimax 1000 IU", tablets by "Eagle Nutritional Inc", (valid up to 02.2022, batch № WJ141).
3. "Detrimax 2000 IU" tablets by "Grocam JBL Sp.z.o.o" (valid up to 11.02.2023, batch № 260220).

All samples were tested within one analytical cycle which allowed to reduce intralaboratory variability of the investigation results. System suitability met the normal values. Chromatograms of both solid and liquid dosage forms did not show peaks interfering with the analysis. Therefore the test results may be considered significant within the established validation characteristics.

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<td></td>
</tr>
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<td>Detrimax 2000 IU 50 μg/tablet</td>
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<td>202.4 μg/g</td>
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On pages 97–98, in the "CONCLUSION" section, mentions of dietary supplements to food "Detrimax Active" and "Detrimax Baby" were removed and the conclusion was changed.

Instead: The method for determination of parameter "Contents of vitamin D₃ (cholecalciferol)" in vitamin dosage forms with HPLC was developed. The method was validated by the following validation parameters: specificity, accuracy, linearity, range, precision. It was shown that the validation results are satisfactory by all specified criteria. The range of the method is 9.5–38 μg/ml.
The online version of the article on the journal's website has been updated.