Erratum: "HPLC-UV Method Development and Validation for Vitamin D_3 (Cholecalciferol) Quantitationin Drugs and Dietary Supplements"

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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 D_3 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 "Pharmia 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 Nutritionals Inc" (valid up to 02.2022, batch № WJ141); "Detrimax 2000 ME", tablets by "Grokam 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 "Pharmia Oy" (valid up to 05.12.2022, batch № 1913870002); "Detrimax 1000 IU", tablets by "Eagle Nutritionals Inc" (valid up to 02.2022, batch № WJ141); "Detrimax 2000 ME", tablets by "Grokam 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 "Pharmia Oy" (valid up to 05.12.2022, batch № 1913870002).
2. "Detrimax Baby", 30 ml, by "Curtis Health Caps Sp.z.o.o" (valid up to 11.2022, batch № 1912004).
3. "Detrimax Active", 30 ml by "Curtis Health Caps Sp.z.o.o" (valid up to 11.2022, batch № 1912041).
4. "Detrimax 1000 IU", tablets by "Eagle Nutritionals Inc", (valid up to 02.2022, batch № WJ141).
5. "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.

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:

- ✓ "Aqudetrim", aqueous solution – 1 g/ml.
- ✓ "Detrimax Baby" – 0.9437 g/ml.
- ✓ "Detrimax Active" – 0.9447 g/ml.

The method error was calculated by equation:

$$\Delta = 1.96 \cdot СКО,$$

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 D_3 (cholecalciferol) in dosage forms (drugs and dietary supplements)**

Drugname	Vitamin D_3 content in the drug (declared)	Vitamin D_3 content in 1 g (1 ml) of the drug (declared)	Vitamin D_3 content in 1 g (1 ml) of the drug (found)	Deviation (found/ declared), %
Ultra-D	25 μg/tablet (tablet weight 425 mg)	58.8 μg/g	52.6 μg/g	–10,5
Detrimax Baby	5 μg/1 drop (200 IU/drop)	5 μg/1 drop, equivalent to 150 μg/ml	87.7 μg/ml	–41,5
Detrimax Active	12.5 μg/1 drop	12.5 μg/1 drop, equivalent to 375 μg/ml	250.3 μg/ml	–33,3
Detrimax 1000 IU	25 μg/tablet	108.7 μg/1 g	106.1 μg/g	–2,4
Detrimax 2000 IU	50 μg/tablet	208.3 μg/1 g	202.4 μg/g	–2,8
"Aquadetrim" aqueous solution	15,000 IU / ml; 1 drop (33.3 μl) = = 500 IU (12.5 μg/33.3 μl) 375 μg/ml	375 μg/ml	370 μg/ml	–1,3
"Akvadetrim" water-soluble tablets	500 IU/tablet (12.5 μg/tablet)	156 μg/1 g	155 μg/g	–0,6

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:

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 "Pharmia Oy" (valid up to 05.12.2022, batch № 1913870002).
2. "Detrimax 1000 IU", tablets by "Eagle Nutritionals 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.

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 ("Aqudetrim", aqueous solution – 1 g/ml).

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The method error for liquid forms (aqueous solutions) was ±5.68 %, for dry dosage forms ±11.37 %, respectively.

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"Aquadetrim" aqueous solution	15,000 IU / ml; 1 drop (33.3 μl) = = 500 IU (12.5 μg/33.3 μl) 375 μg/ml	375 μg/ml	370 μg/ml	–1,3
"Akvadetrim" water-soluble tablets	500 IU/tablet (12.5 μg/tablet)	156 μg/1 g	155 μg/g	–0,6

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 D_3 (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 results of the method validation and investigation of actual samples may be used for determination of vitamin D_3 in vitamin products based on water soluble substances of vitamin D_3, as aqueous solutions and solutions of fatty acid triglycerides.

Based on the assay results, it was established that:

1. Actual contents of vitamin D_3 for product "Aquadetrim", aqueous solution 10 ml by JSC "Medana Pharma" (valid up to 04.2023, batch № 050420), Actual contents of the vitamin was 370 μg/ml, and the deviation of the actual contents from the label claim was –1.3 %.
2. Actual contents of vitamin D_3 for "Aquadetrim", water soluble tablets by JSC "Akrikhin" (valid up to 04.2022, batch № 170420) was 155 мкг/г, and the deviation of the actual contents from the label claim was –0.6 %.
3. Actual contents of vitamin D_3 for biologically active dietary supplement "Ultra-D", chewable tablets by "Pharmia Oy" (valid up to 05.12.2022, batch № 1913870002) 58,8 μg/g, and the deviation of the actual contents from the label claim –10,5 %.
4. Actual contents of vitamin D_3 for biologically active dietary supplement "Detrimax Baby", 30 ml, by "Curtis Health Caps Sp.z.o.o" (valid up to 11.2022, batch № 1912004), 93 μg/ml, and the deviation of the 93 μg/ml, and the deviation of the actual contents from the label claim –41.5%.
5. Actual contents of vitamin D_3 for biologically active dietary supplement "Detrimax Active", 30 ml by "Curtis Health Caps Sp.z.o.o" (valid up to 11.2022, batch № 1912041) was 265 μg/ml, and the deviation of the actual contents from the label claim –33.3 %.
6. Actual contents of vitamin D_3 for biologically active dietary supplement "Detrimax 1000 IU", tablets by "Eagle Nutritionals Inc" (valid up to 02.2022, batch № WJ141) was 106.1 μg/g, and the deviation of the actual contents from the label claim –2.4 %.
7. Actual contents of vitamin D_3 for biologically active dietary supplement "Detrimax 2000 IU", tablets, by "Grocam JBL Sp.z.o.o." (valid up to 11.02.2023, batch № 260220) was 202.4 μg/g, and the deviation of the actual contents from the label claim –2.8 %.

It should be established that contents of vitamin D_3 in vitamin dietary supplements tend to a greater dispersion from the declared one. Liquid dietary supplements based on fatty acid triglycerides require comprehensive control of raw materials used (control of peroxide and acid value) as vitamin D_3 is unstable at the light and oxygen-rich medium.

Corrected to: The method for determination of parameter "Contents of vitamin D_3 (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 results of the method validation and investigation of actual samples may be used for determination of vitamin D_3 in vitamin products based on water soluble substances of vitamin D_3 and as aqueous solutions.

Based on the assay results, it was established that:

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2. Actual contents of vitamin D_3 for "Aquadetrim", water soluble tablets by JSC "Akrikhin" (valid up to 04.2022, batch № 170420) was 155 мкг/г, and the deviation of the actual contents from the label claim was –0.6 %.
3. Actual contents of vitamin D_3 for biologically active dietary supplement "Ultra-D", chewable tablets by "Pharmia Oy" (valid up to 05.12.2022, batch № 1913870002) 58,8 μg/g, and the deviation of the actual contents from the label claim –10,5 %.
4. Actual contents of vitamin D_3 for biologically active dietary supplement "Detrimax 1000 IU", tablets by "Eagle Nutritionals Inc" (valid up to 02.2022, batch № WJ141) was 106.1 μg/g, and the deviation of the actual contents from the label claim –2.4 %.
5. Actual contents of vitamin D_3 for biologically active dietary supplement "Detrimax 2000 IU", tablets, by "Grocam JBL Sp.z.o.o." (valid up to 11.02.2023, batch № 260220) was 202.4 μg/g, and the deviation of the actual contents from the label claim –2.8 %.

As a result of the studies, it was shown that for the determination of vitamin D_3, used in the form of a water-soluble substance, in medicines and dietary supplements for food, it is possible to use methods without carrying out the saponification stage. This provides tangible benefits, since allows you to avoid the loss of the active substance in the process of multi-stage sample preparation, because vitamin D_3 is unstable in the light and in an environment rich in oxygen.

The online version of the article on the journal's website has been updated.