

ISO 13779-3:2018-12 (E)

Implants for surgery - Hydroxyapatite - Part 3: Chemical analysis and characterization of crystallinity ratio and phase purity

Contents	Page
Foreword	v
Introduction	vi
1 Scope	1
2 Normative references	1
3 Terms and definitions	1
4 Chemical analysis	4
4.1 General	4
4.2 Analytical methods	4
4.3 Apparatus for chemical analysis	5
4.4 Reagents for chemical analysis	5
4.5 Procedure	5
4.6 Expression of results	6
5 X-ray diffraction analysis	6
5.1 General	6
5.2 Apparatus	6
5.3 Preparation of test samples	7
5.3.1 General	7
5.3.2 Coatings	7
5.3.3 Bulk sample	7
5.4 Calibration specimens	7
5.5 X-ray diffraction pattern collection	7
5.5.1 General	7
5.5.2 Identification of the crystallized phases	8
5.6 Calibration curves, limits and uncertainties	8
5.6.1 General	8
5.6.2 Plotting the calibration curves for the foreign phases	8
5.6.3 Detection limit (DL) and quantification limit (QL) of foreign phases	9
5.6.4 Uncertainty for determination of foreign phases content	10
5.7 Qualitative and quantitative determination of the foreign phases	11
5.7.1 Procedure	11
5.7.2 Expression of results	11
5.8 Calcium to phosphorous (Ca:P) ratio determination	11
5.8.1 General	11
5.8.2 Measurements on the sample	11
5.8.3 Uncertainty of Ca:P measurement	12
5.8.4 Expression of results	12
5.9 Determination of the crystallinity ratio	13
5.9.1 Rationale on different methods of determination of the crystallinity ratio	13
5.9.2 General	13
5.9.3 Procedure A	13
5.9.4 Procedure B	14
5.9.5 Uncertainty of the crystallinity ratio	14
5.9.6 Expression of results	14
6 Test report	14

Annex A (informative) Contamination of calcium phosphate	16
Annex B (normative) Testing of the purity of the phases used in the production of the calibration curves	17
Annex C (informative) Examples of X-ray diffraction patterns collected from various mixtures used to plot the calibration curves	19
Annex D (normative) Positions of hydroxyapatite lines used to measure the crystallinity ratio	24
Annex E (normative) Methods for the preparation of reference materials	25
Annex F (informative) Uncertainty calculation of Ca:P ratio	28
Annex G (informative) QuantitativePhaseAnalysis(QPA)byRietveldRefinementofXRDData	30
Annex H (informative) Alternative method: Determination of the crystallinity ratio	31
Bibliography	32