Record Nr. UNINA9910511424103321 NMR spectroscopy in pharmaceutical analysis [[electronic resource] /] / **Titolo** edited by Ulrike Holzgrabe, Iwona Wawer, Bernd Diehl Pubbl/distr/stampa Oxford, : Elsevier, 2008 **ISBN** 1-281-76266-0 9786611762667 0-08-095152-X 0-08-055819-4 Descrizione fisica 1 online resource (525 p.) Altri autori (Persone) HolzgrabeU (Ulrike) Wawerl (Iwona) DiehlB (Bernd) 615.19 Disciplina 615.1901 Soggetti Nuclear magnetic resonance spectroscopy Drugs - Analysis Electronic books. Lingua di pubblicazione Inglese **Formato** Materiale a stampa Livello bibliografico Monografia Note generali Description based upon print version of record. Nota di bibliografia Includes bibliographical references and index. Front Cover; NMR Spectroscopy in Pharmaceutical Analysis; Copyright Nota di contenuto Page; Table of Contents; Preface; List of Contributors; List of Editors; Part I FUNDAMENTALS AND TECHNIQUES: Chapter 1 Principles in NMR Spectroscopy; 1 Short History; 2 The NMR Experiment; 2.1 Excitation, relaxation and sensitivity; 2.2 Relaxation; 3 Chemical Shift; 3.1 Electronic density; 3.2 Anisotropy; 3.3 Mesomerism; 3.4 Steric effects; 4 Calibration and Relative Scale ppm; 5 Spin-Spin Coupling; 5.1 The coupling constant; 5.2 Multiplicity; 5.3 Roof effect; 5.4 Angular dependence of the coupling constant 5.5 Heteronuclear coupling 5.6 13C NMR satellites in 1H NMR spectra; 6 Heteronuclear Spectra; 6.1 Decoupling; 6.2 Quantitative heteronuclear NMR: 7 Molecular Dynamics: 7.1 Deuterium exchange and solvent effects; 8 Chemical Derivatisation; 9 Stereochemistry; 9.1 Diastereomerism; 9.2 Atrop or axial chirality; 9.3 Enantiomeric excess;

9.4 Diastereotopy: 10 Two-dimensional Methods for Structure

Elucidation; 11 Experimental Data; Chapter 2 Quantitative NMR in the Solution State NMR: 1 Introduction: 2 Basics: 3 Quantitative NMR Spectroscopy; 3.1 Relative method; 3.2 Absolute method; 4 Validation 4.1 Linearity4.2 Robustness; 4.3 Specificity and selectivity; 4.4 Accuracy; 4.5 Precision; 4.6 Measurement uncertainty; 4.7 Round robin tests; 5 Sensitivity; 6 Conclusion; Chapter 3 qNMR in Solid State; 1 Introduction to Solid-state NMR; 2 Quantitative Aspects of Solid-state NMR, Protocol for Quantitation; 3 An Example: Quantitation of Pseudoephedrine in Dosage Form: 4 Quantitation of Polymorphs and Formulated Drugs: Chapter 4 Microcoil Nuclear Magnetic Resonance Spectroscopy; 1 Introduction; 2 Intrinsic NMR Sensitivity; 3 Development of Small Coils for High-Resolution NMR 3.1 Saddle/Helmholtz coils3.2 Solenoidal coils; 3.3 Planar RF coils; 3.4 Novel microcoil designs; 3.5 High-temperature superconducting microprobes: 4 Sensitivity Comparisons: 5 Nanoliter Volume Applications of RF Microcoils - Hyphenated cITP-NMR; 6 Microliter Volume Applications of RF Microcoils; 6.1 Helmholtz/saddle coils; 6.2 Solenoidal coils; 6.3 Superconducting microcoils; 7 Hyphenation of Microseparation Techniques with Microliter NMR Detection; 8 Multiple Coil Probeheads; 9 Solid-State Applications of Small Coils; 10 Conclusion

Chapter 5 qNMR Spectroscopy in Drug Analysis - A General View1 Introduction; 2 NMR Spectroscopy in International Pharmacopoeias; 2.1 Identification of drugs; 2.2 Tests; 2.3 Assay; 3 Validation; 4 Conclusions; Part II GENERAL APPLICATIONS; Chapter 1 Investigation of Multi-Component Drugs by NMR Spectroscopy; 1 Introduction; 1.1 Solvent; 1.2 pH value; 1.3 Temperature; 1.4 Auxiliary reagents; 1.5 Limitations; 2 Codergocrine Mesylate; 2.1 1H NMR spectroscopy; 2.2 13C NMR spectroscopy; 2.3 High-performance liquid chromatography; 2.4 HPLC versus NMR spectroscopy Chapter 2 NMR Applications for Polymer Characterisation

Sommario/riassunto

For almost a decade, quantitative NMR spectroscopy (qNMR) has been established as valuable tool in drug analysis. In all disciplines, i. e. drug identification, impurity profiling and assay, qNMR can be utilized. Separation techniques such as high performance liquid chromatography, gas chromatography, super fluid chromatography and capillary electrophoresis techniques, govern the purity evaluation of drugs. However, these techniques are not always able to solve the analytical problems often resulting in insufficient methods. Nevertheless such methods find their way into international pharm