

<<纳米科学与技术大全5>>

图书基本信息

书名：<<纳米科学与技术大全5>>

13位ISBN编号：9787030346605

10位ISBN编号：7030346602

出版时间：2012-7

出版时间：科学出版社

作者：戴维 L.安德鲁斯

页数：547

字数：901500

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<<纳米科学与技术大全5>>

内容概要

纳米科学与技术大全5：自组装与纳米化学（导读版）作者将生态学不同领域的理论和实验进展与新方法相结合，在个体层次与群落结构、生态系统功能间，微观世界与宏观生态间的融合做了很好的尝试，为我们理解生态现象、生态过程以及生态功能展示了一个很好的途径。

纳米科学与技术大全5：自组装与纳米化学（导读版）可作为研究生生态课程的参考书，对于从事生态学教学和研究的教师和科研人员及生态学者有非常高的参考价值。

<<纳米科学与技术大全5>>

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章节摘录

版权页：插图：5.02.4.2 Direct Probing of Ligands To address the NP ligands more directly, two techniques have been recently applied in the literature: synchrotron X-ray photoelectron spectroscopy (XPS) and solution NMR [34,35]. With XPS, the various elements present in the nanocrystal core and on the surface can be probed and information on their local environment is obtained. For instance, in the case of PbS, Lobo and coworkers observed a set of S- and Pb emission peaks which they could attribute to various surface species, either unpassivated, linked to a specific ligand, or oxidized [34]. A closely related technique that has been used to identify NP ligands is Rutherford backscattering spectroscopy (RBS) [34,35]. This experiment does not provide information on the exact location of the atoms of interest (in the core or on the surface) but nevertheless, the presence of, for instance, phosphorous atoms in the sample can be taken as evidence for TOP ligands. Both XPS and RBS are ex situ analysis techniques, carried out on nanocrystal solids. This means that traces of unreacted precursors or free ligands might lead to spurious results, since they can be hardly distinguished from bound ligands. Therefore, a careful assessment of the sample purity is crucial in these measurements. In addition, little information on the ligands is obtained, and a study of their dynamical behavior in solution is not possible. The drawback of ex situ analysis can be overcome by solution NMR. This has been applied to the study of NP suspensions for more than 15 years, yet it has mainly been limited to one-dimensional (1D) ^1H NMR. In that case, NMR raises similar problems of interpretation as XPS and RBS, discrimination between nanocrystal ligands and free molecules is not straightforward. Nevertheless, solution NMR has the advantage of being an in situ technique that involves only a minimal perturbation of the sample, and many early studies at least suggested that NP ligands can be observed with NMR. In the remainder of this chapter, we discuss how the various pitfalls of ligand identification can be overcome with solution NMR, and show that it provides much more information on the NP ligands than a positive ligand identification only.

5.01.5.1 Solution NMR Techniques for Observing QD Ligands

5.02.5.1.1 A brief introduction in solution NMR spectroscopy

With solution NMR spectroscopy, one investigates molecular species dissolved or suspended in a liquid sample. Only atoms that carry a nuclear spin can be observed, and only isotopes with a nuclear spin $1/2$, such as ^1H , ^{13}C , ^{15}N , and ^{31}P , are considered here as they afford high-resolution spectra to be obtained. We will explain the technique for ^1H , yet the concepts apply to the other nuclei as well. The sample is placed in a homogeneous magnetic field B_0 , directed along the z-axis. This field lifts the spin degeneracy of, in the case of protons, the two possible spin states (up and down). The energy difference is small, yet it results in a slight excess of spin-up protons producing a net longitudinal magnetization M along the z-axis of B_0 (Figure 12(a)). The phase of the precessing spins remaining randomly distributed with respect to the xy plane, no net transverse magnetization is generated. Next, a radio-frequency magnetic field pulse B_1 , along the x-axis, is used to rotate M by 90° into the xy plane, here along the y-axis (Figure 12(b)).

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