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Magnetic Resonance Studies of Ion Solvation. The Coordination Number of Gallium(III) Ions in Aqueous Solutions

Sir:

An extensive ^{17}O nuclear magnetic resonance study of aqueous solutions containing Ga^{3+} ions has been carried out. Herein are reported the results of the coordination number determinations.[1]

Two independent methods were used to determine the number of water molecules in the sphere of hydration of the Ga^{3+} ions. The coordination number was determined from the ratio of areas of the bound to the free water, where one is shifted relative to the other by the addition of Co^{2+} ions, and also was determined from a measurement of the shift of the free water relative to reference pure water.[2-4]

To pure water containing about 10% ^{17}O and acidified with perchloric acid four successive amounts of cobaltous perchlorate were added. The paramagnetic shift of the ^{17}O of the water molecules was measured relative to a pure water reference. The following results obtained are given in Table I. Extrapolation of these results to 1 mole of Co^{2+}/55.5 moles of H_2O gives 11000 cps, corresponding to 1905 ppm.

Table I[a]

Detmn	n_{H_2O}	n_{HClO_4}	$n_{Co(ClO_4)_2}$	$\Delta f_{ref/sol}$, cps
1	59.64	0.95	0.0598	600
2	60.13	0.95	0.1303	1320
3	60.59	0.95	0.1973	1972
4	60.96	0.95	0.2498	2615

[a] Amounts expressed in millimoles; the four points were on a straight line.

To solution 4, 0.5805 g of $Ga(ClO_4)_3 \cdot (9.38 \pm 0.23) H_2O$ was added. The measured paramagnetic shift for this solution was 2370 cps, whereas the calculated is 2142 cps. Assuming that the increase in the observed shift relative to the calculated is caused by the Ga^{3+} ions retaining part of the water in the hydration sphere, the coordination number is calculated to be 6.28 ± 0.26.

For the same solution the ratio of the areas of the absorption signals of the bound to the free water yielded a coordination number of 5.89 ± 0.20 (as an average of four signals).

The two methods are seen to be in good agreement. They are, however, sensitive to different factors. The method, based on the measurement of the shift itself, requires accurate determination of the water of hydration of the salt. As a matter of fact the error in the coordination number of the ion in the solution will be the error in the determination of the hyration of the salt. On the other hand, the method based on the ratio of the areas of the free to the bound water signals will not require this. The advantage of the method based on the measurement of the shift is that it is easier to measure the shift of the water in bulk than to measure areas. In particular the measurement of the area of the bound water to the desired precision—accurately enough to determine the hydration numbers—is very elaborate. Depending on the circumstances one of the two methods should be preferred. In the case of the Ga^{3+} ion the experimental error of the two methods came out to be about the same.

As given above, the paramangetic shift of the free water relative to the pure water was found to be 2370 cps. The shift of the free water relative to the bound was 2190 ± 10 cps. Hence there is a paramagnetic shift of 180 cps of the bound water relative to the pure.

The width of the bound water signal at 35° was 753 cps. The width of the free water signal was 580 cps.

(5) Work performed at the Lawrence Radiation Laboratory, University of California, Berkeley, Calif.

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Complete Sequence of Biosynthesis from *p*-Hydroxybenzoic Acid to Ubiquinone[1]

Sir:

A complete biosynthetic sequence can now be formulated for the pathway from *p*-hydroxybenzoic acid (I) to ubiquinone (IX). Since *p*-hydroxybenzoic acid is a precursor for ubiquinone in various microorganisms[2] and in animals,[2-4] this sequence may be generic for many species of life utilizing ubiquinone in electron transfer of respiration and coupled phosphorylation. However, the diversity of life utilizing ubiquinone may signify that pathways alternative to this sequence will be found.

Four new quinones, apparent precursors of ubiquinone, have been isolated by extensive fractionation of a lipid extract from *Rhodospirillum rubrum*. Structural studies show these products to be 2-decaprenyl-6-methoxy-3-methyl-1,4-benzoquinone (VII, $n = 10$),

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