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Short Communication

A Convenient Method for the Preparation of Nitrilotriacetic Acid-d_9

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Nitrilotriacetic acid [$N(CH_2COOH)_3$,NTA] is a versatile and useful ligand of great environmental concern and a number or reports on its complexes have appeared in recent years.[1] As a part of a study of Ti(III)-NTA complexes we needed NTA-d_9 of high isotopic purity, but to our surprise no synthetic procedure was available in the literature. Standard base- or acid-catalyzed exchange techniques yielded no exchange of the non-labile protons of either NTA or its triethyl ester. A number of preparations of glycine-d_5 have been presented, and 28 and 70–100% deuteriation, respectively, was observed upon treatment of glycine with platinum black and D_2O.[2] Using the procedure described below we obtained NTA-d_9 with an isotopic purity of >99.8, i.e., only traces of NTA-d_8 and no traces of other products could be detected by careful examination by mass spectroscopy.

Experimental

Materials. $PtO_2 \cdot xH_2O$ (Janssen, 99.99%) was stirred with D_2O and evaporated three times prior to use. NTA (Jansen, 99%) and D_2O (CIL, 99.9% D) were used as received.

Instrumentation. Mass spectra were recorded on a VG Quattro II instrument equipped with ESPC electrospray.

Preparation of NTA-d_9. NTA (1.0 g), PtO_2 (0.1 g) and 10 ml D_2O were mixed in a tight Teflon vessel and placed in a steel bomb which was heated to 150 °C for 30 h. The catalyst was filtered off and the solvent removed by evaporation. After two repetitions an essentially quantitative yield of the completely exchanged product was obtained. Recrystallization from D_2O yielded crystals of NTA-d_9 of >99.8% isotopic purity.

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