STEROIDS

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For known compounds, indicate the observed and literature melting points for crystalline solids, and/or the observed and literature optical rotations, in the following formats: mp xx oC (lit [ref] xx oC) and/or \[\text{[D}^C \text{xx oC (c, xx g/100 mL; solvent)} (\text{lit [ref]}\text{[D}^C \text{xxo (c, xx g/100 mL; solvent)}. Provide comments (either in general or for individual compounds) comparing the observed $^1$H and $^{13}$C NMR data of known compounds with the literature values, e.g., The $^1$H and $^{13}$C NMR data (xx MHz, solvent) agreed well with the literature values [ref]. It is not necessary to report complete data sets for known compounds. However, significantly different or improved data (e.g., different chemical shifts, data in different solvents, data taken at higher field, improved coupling analyses, etc.) should be reported in the Experimental Section or in a Table of NMR data and assignments.

Many naturally occurring steroids are isolated as glycoside derivatives. In such cases, the structures and absolute configuration of the individual sugar residues should be determined after hydrolysis. The absolute configuration of an individual sugar can be determined by comparing its optical rotation with the literature value for sugars of well-established absolute configuration. Another approach is direct comparison with authentic samples of the D and L sugars, or their derivatives, by high-pressure liquid chromatography (HPLC) or by gas chromatography (GC) on columns containing suitable chiral adsorbents, provided it is demonstrated that the enantiomeric compounds give separate peaks on the chiral columns.

Criteria for the purity of all compounds and of compounds with biological data: All new compounds need to be pure. Evidence of high purity is essential where biochemical or biological assay data are presented and related to compound structures; these compounds are termed "SAR compounds." The purity of SAR compounds should be more than 98 percent; the purity of other compounds should be more than 95 percent. Any questions regarding the purity of SAR compounds should appear in the Results.

The methods used to establish the purity of steroids subjected to biochemical or biological assays must be described in the Experimental Section. Most steroids obtained in pure form will be crystalline. Thus, there should be an attempt to purify and crystallize all products of chemical reactions or compounds isolated from plant extracts. Melting points should be recorded and reported for all crystalline compounds. It is strongly recommended that optical rotations be reported for new compounds. The weights and % yields should be reported for products isolated from chemical transformations.

Evidence for purity can take many forms: Combustion analyses for carbon, hydrogen and nitrogen are adequate. These data should appear in the Experimental Section and should agree with the calculated data within 0.4 percent. A recommended form for presentation is: C18H23NO4: calcd. C, 68.12; H, 7.30; N, 4.41. Found. C, 68.50; H, 7.18; N, 4.26. When satisfactory combustion analyses are not available, evidence of purity should be provided by HPLC chromatograms run in two divergent solvent systems (typically normal and reversed phase) or by high quality proton NMR spectra obtained at high signal-to-noise. These chromatograms or spectra should be included as supplementary data.

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