SUPPLEMENTARY INFORMATION

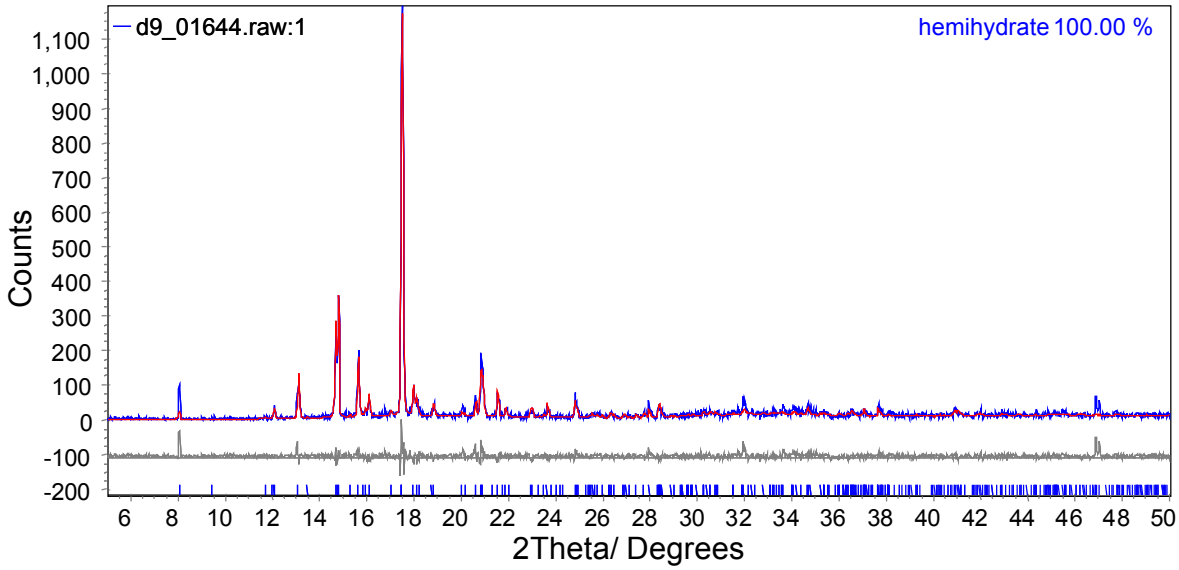

Figure 1. Rietveld fitting of the observed diffractogram for the hemihydrate of androsterone with the calculated powder pattern. The sample was examined on a silicon slide after grinding under liquid nitrogen.

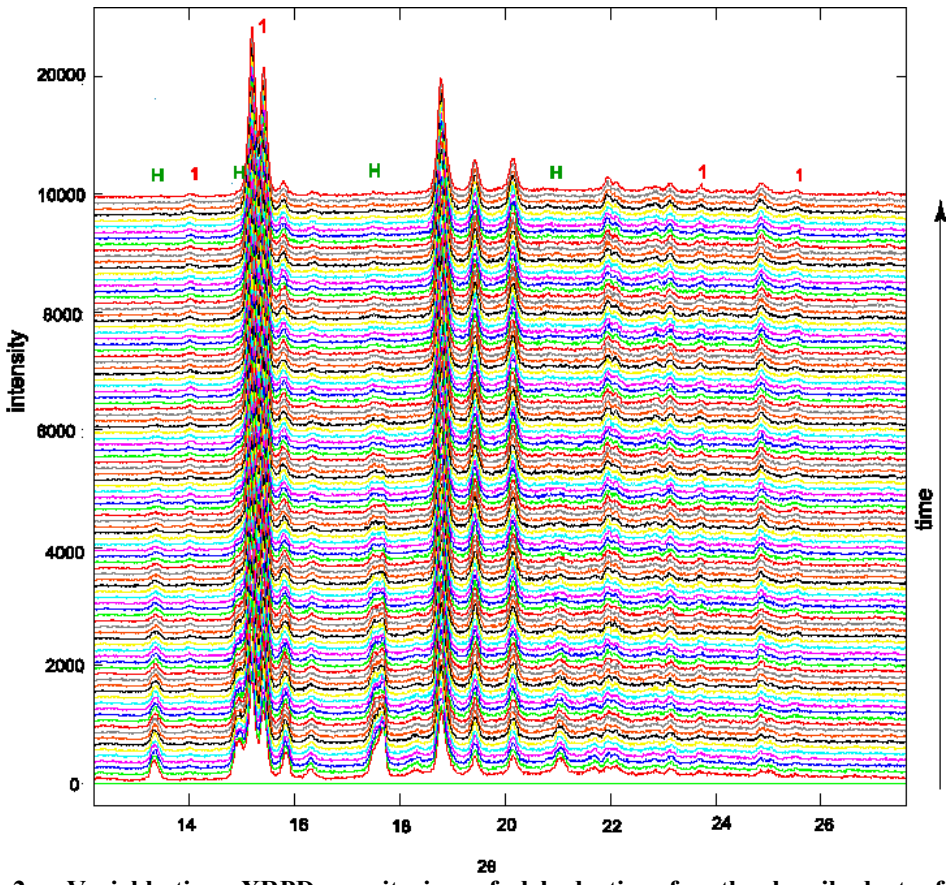

Figure 2. Variable-time XRPD monitoring of dehydration for the hemihydrate form of androsterone. Peaks due to the hemihydrate (H) and form 1(1) are labelled.

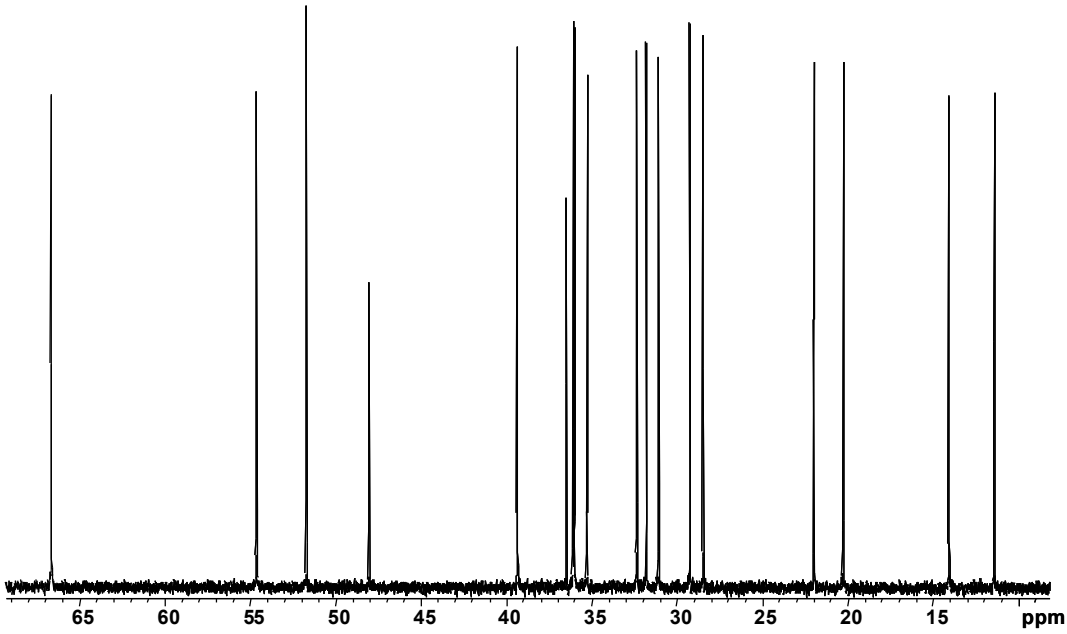

Figure 3. Androsterone solution-state ^{13}C NMR spectrum

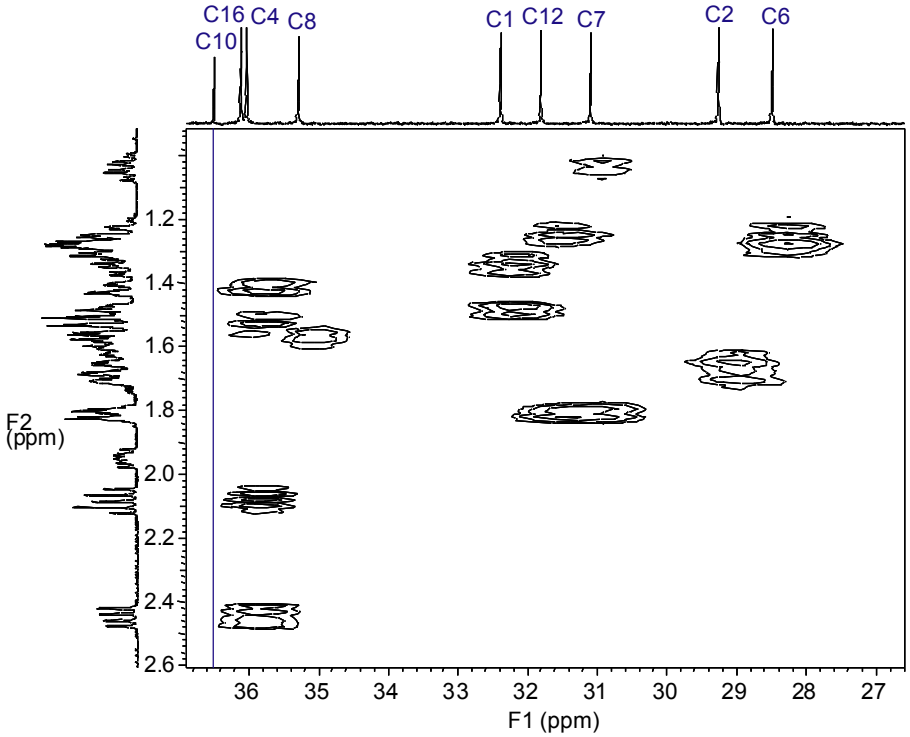

Figure 4. Androsterone HSQC spectrum, showing that the C10 signal does not correlate with any proton peak.

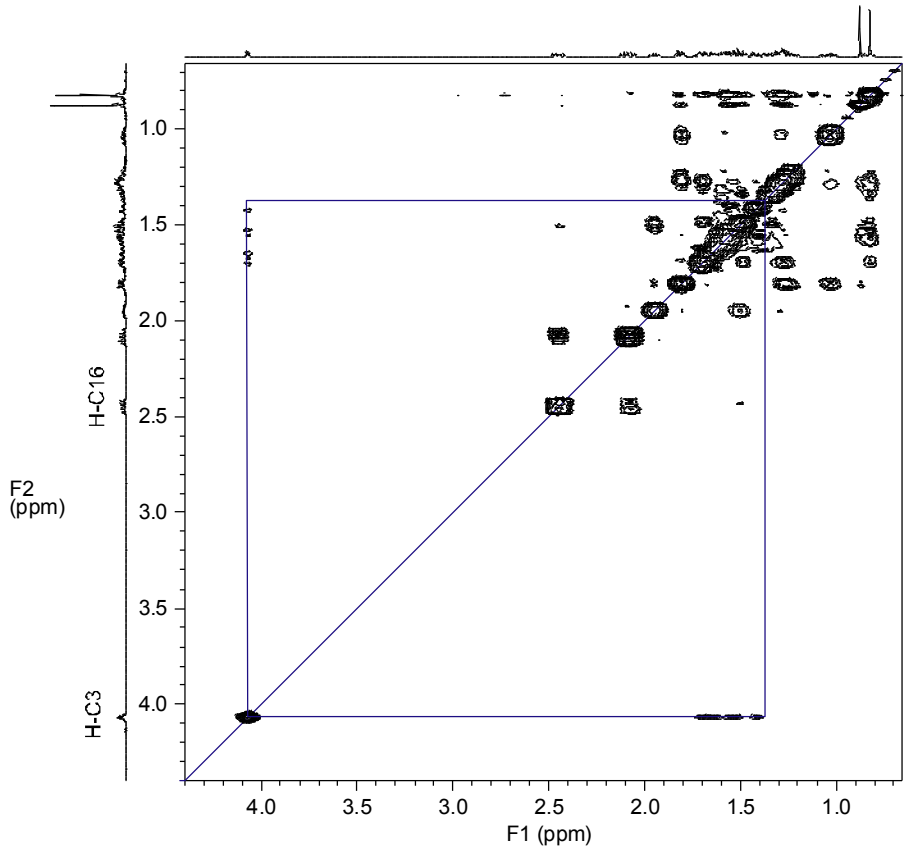

Figure 5. Androsterone NOESY spectrum, showing that the proton signal for C3 (4.1 ppm) correlates with peaks for protons other than C16 protons (1.5-2.0 ppm).

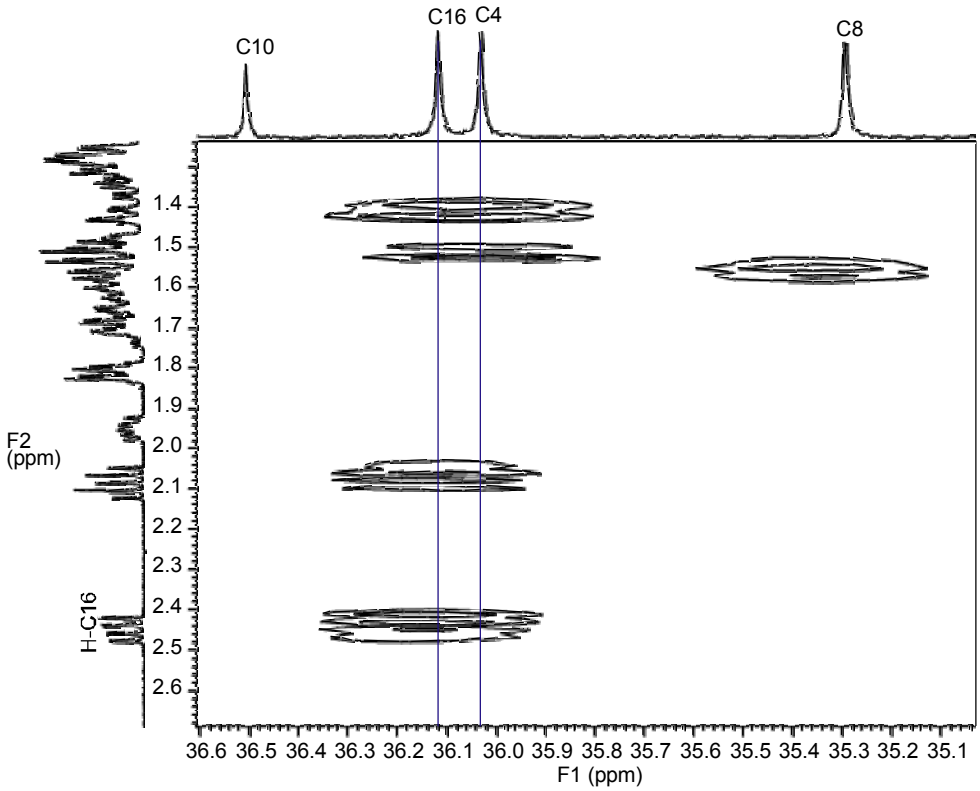

Figure 6. Androsterone HMBC spectrum (zoomed-in scale), showing that the signal for C4 does not correlate with peaks from protons which correlate with the C3 proton peak in the NOESY experiment. The C16 peak correlates with that for the deshielded proton at C16.

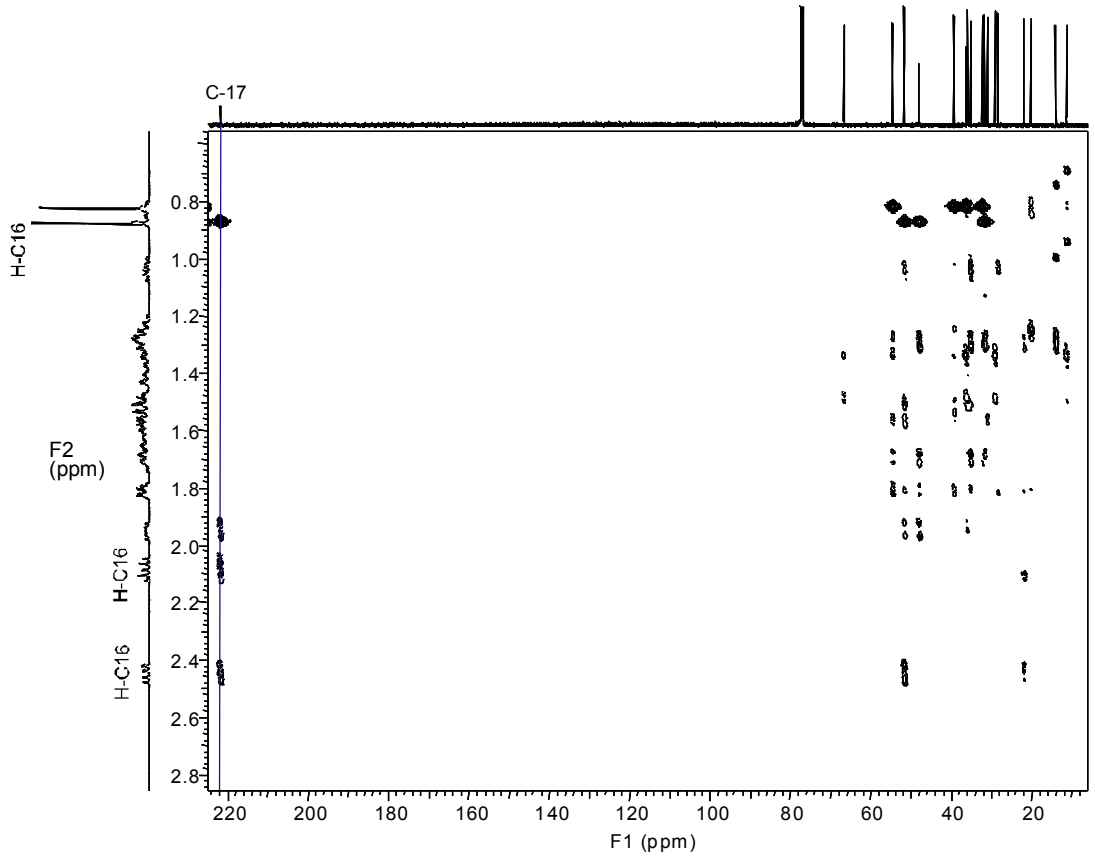

Figure 7. Androsterone HMBC spectrum, showing that the signals for the protons at C16 correlate with the C17 (carbonyl) peak.