

The content of the spiro-dienone structure was found to be over 3% in MWL1 by quantitative ¹³C NMR, as abundant as the coniferaldehyde in this spruce lignin sample. The whole aromatic and ethylenic carbon region has an integral of 6.12. The baseline is flat as indicated by a integral on the baseline close to the signal B4.



Unambiguous assignment of spiro-dienone NMR signals by combination of QUAT, HMBC and HSQC NMR spectra.

In QUAT spectrum, the methoxy signals at 56.3 ppm was successfully suppressed. The dienone carbonyl (B4) carbon at 180.7 ppm and the B1 quaternary carbon at 55.5 can be seen clearly. Some positive noise from residual CH_2 signals and some negative noise from residual CH signals are present, but they do not affect the assignment.

HMBC confirmed the long-range connections of the dienone ring B.



HSQC-TOCST of side chain region showing the connection of spiro side chain CHn groups



HSQC-TOCSY of aromatic region showing the connection between CH-B5 and CH-B6.