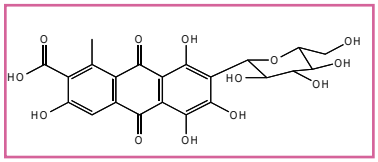
Application of qNMR. Part 2: Quantification of carminic acid in cochineal extract

NMR spectroscopy, in principle, counts the number of atoms in molecules, and so the purity of the sample can be quantified from the ratio of the signal intensities of a target material and a reference. Quantitative NMR (qNMR) is widely applicable because the target materal is not used as a standard sample of known purity. This Note introduces an example of qNMR analysis for carminic acid in cochineal extract, the standard sample of which is not easily available.

Carminic acid



Carminic acid is a main component of cochineal extract, originally obtained by drying scale insects and extracting them with ethanol or water.

Purpose: The purity of test reagents of carminic acid and that of food additives of cochineal extract are examined by qNMR.

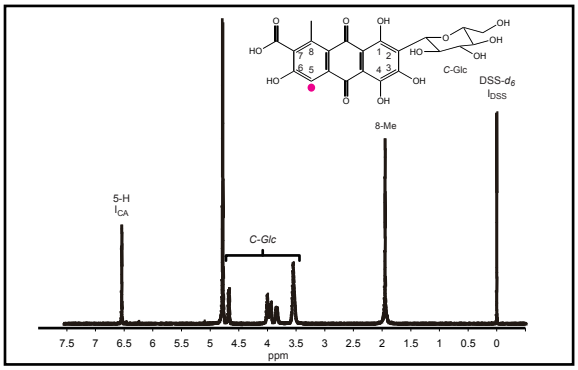


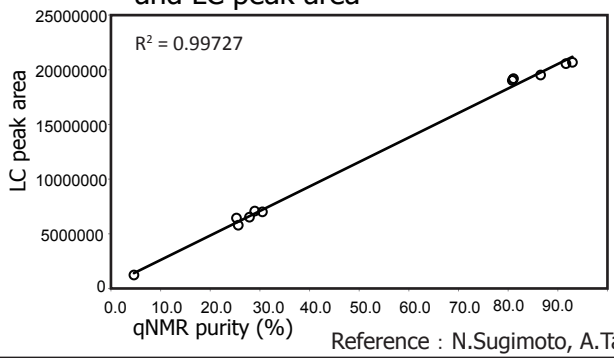
Fig. 1 qNMR spectrum of carminic acid (Spectrometer: JNM-ECA600)

Table Summary of sample information and results of qNMR analyses

Samples	Purity in catalog (%)	LC peak area* (UV 490nm)	qNMR purity (%)**	qNMR purity (%)*** (as potassium hydrate)
Carminic acid				
1 (high purity sample)	-	20682647	81.8	97.1
2 (reagent)	>70%(HPLC)	6439525	21.3	25.3
3 (reagent)	>95%(Spectrophotometric)	19189340	78.3	92.9
4 (reagent)	-	21172301	68.3	81.1
5 (reagent)	-	19028131	68.1	80.8
6 (reagent)	95%	20561664	77.2	91.6
7 (reagent)	70～90%	19521465	72.9	86.5
8 (reagent)	-	7079698	24.4	29.0
Cochineal extract				
9 (food additive powder)	-	5785440	21.6	25.6
10 (food additive powder, low allergen)		6519563	23.5	27.9
11 (food additive powder)	-	7006552	25.7	30.5
12 (food additive liquid)	-	1232696	3.9	4.6

* Peak of 18.2min retention time in HPLC anaysis.
** Calculated from the signal of carminic acid 5H●.
*** Obtained from X-ray crystal structure analysis.

Fig. 2 Correlation beween qNMR purity and LC peak area



Conclusion

1. Purities of carminic acid in reagents and cochineal extracts were determined by qNMR.

2. Purities from qNMR were confirmed to have strong correlation with LC peak area.

It was found that qNMR is a useful quantification analysis for natural products, for which standard samples for quantification are not available.

Reference : N.Sugimoto, A.Tada, T. Suematsu et al. Food Hyg. Saf. Sci., 2010, 51, 19-27.

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