*Low-Field NMR*

# An Innovative Technique to Measure and Monitor the Moisturizing Efficacy of Cosmetic Actives on the Hair Fiber

NMR (Nuclear Magnetic Resonance) methods developed by Cerevaa enable cosmetic industry players, including Mibelle Biochemistry, to scientifically demonstrate the moisturizing efficacy of their active ingredients. As an added benefit, the reparative efficacy of actives on the hair fiber can also be assessed. The effects of the active PinoPlex are discussed.

## THE EXPERTS

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### Understanding Low-Field NMR and Hair Hydration Mechanisms

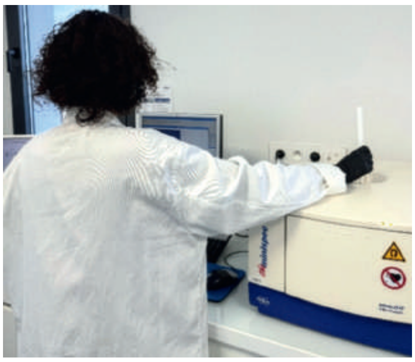
Nuclear Magnetic Resonance (NMR) methods are widely used in fundamental and academic research but remain relatively underutilized in industrial settings. For the past 20 years, Cerevaa has taken on the challenge of making this innovative technology accessible by developing applied methodologies tailored to the economic constraints of the cosmetics sector.

In this context, measuring the hydration of keratin-based materials—whether hair fibers or skin tissue—is a key parameter for assessing tissue health. For hair, *in vivo* testing on volunteers enables the evaluation of various criteria under real-use conditions to determine the relevance and efficacy of a given treatment.

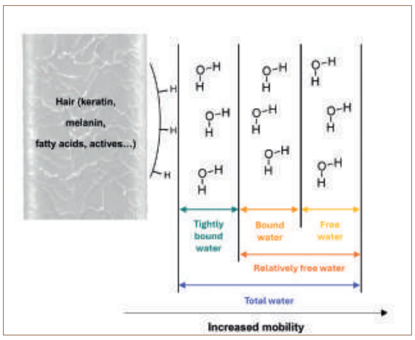
However, it is essential to go further and scientifically demonstrate how an active ingredient interacts with the hair fiber to deliver moisturizing and/or reparative benefits.

Low-field NMR relaxometry methods (20 MHz) (Figure 1) make it possible to extract various informative NMR parameters. When combined with proprietary calculation methods developed by Cerevaa, these parameters provide insight into the water content within the hair fiber and the structural organization of this water within the fiber.

As a reminder, NMR enables the detection and quantification of signals emitted by atomic nuclei, including proton signals (hydrogen nuclei). When subjected to a magnetic field and stimulated by a radiofrequency pulse, these nuclei respond differently depending on their molecular environment. This makes it possible to obtain information not only on the total water content of the sample but also on its compartmentalization (relative proportions of tightly bound water, bound water, and ►►►



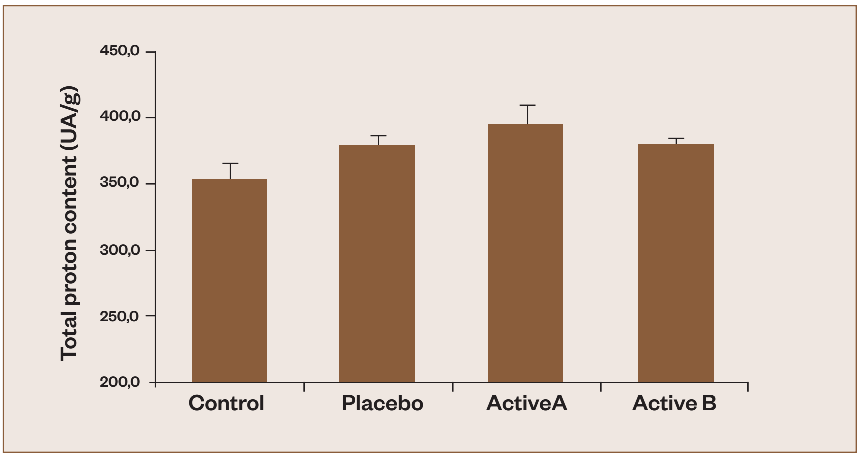
▲ **Figure 1:** Low-field NMR measurements.



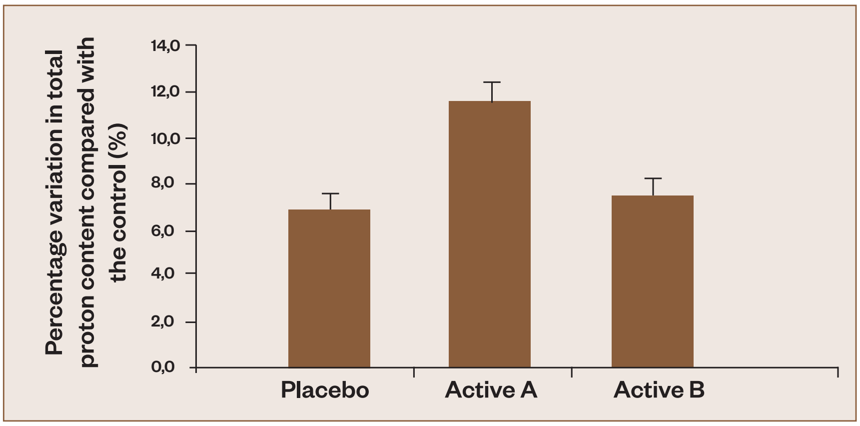

▲ **Figure 2:** Schematic representation of water compartmentalization measured by low-field NMR.

▶▶▶ free water). In parallel, proton mobility reflects the interaction forces between water protons and their surrounding environment, governing how water molecules bind and structure themselves (Figure 2).
Under controlled and defined conditions, it is therefore possible to access detailed information on both the water content within the hair and the structural organization of this water.

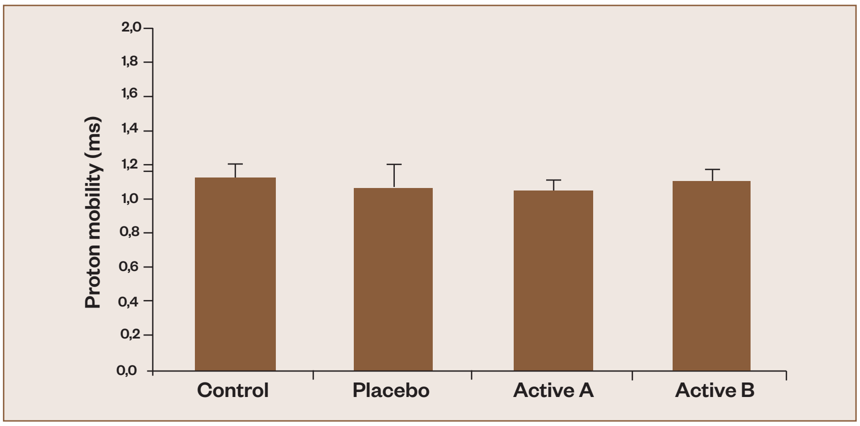
**From Measurement to Demonstration of Efficacy**
The methodology developed by Cerevaa is designed to be both simple and efficient. All hair tresses are standardized prior to analysis through washing with a mild shampoo. After treatment with the active ingredient or formulation, according to specifications defined in collaboration with the industrial partner, NMR analyses are performed directly on the tresses. These tresses may vary in type (Caucasian, curly, coily, etc.) and condition (natural, sensitized, heat-stressed, etc.). Measurements can also be conducted with or without a rinsing phase. By comparing the results with untreated control tresses, valuable insights into treatment efficacy can be obtained, both in terms of hydration and repair, particularly for sensitized or textured hair.
Work carried out in collaboration with Mibelle Biochemistry on two active ingredients illustrates the applications of this methodology. The objective was to assess both the moisturizing and reparative efficacy of each active on hair tresses damaged by bleaching.
Both active ingredients were tested at a 1% dilution in water to reflect typical use conditions in finished products.
NMR analyses were performed under the following conditions on damaged and standardized hair tresses:
• untreated control hair;
• hair treated with placebo (= water);
• hair treated with active A (PinoPlex);
• hair treated with active B
Figures 3a and 3b show, respectively, the total proton content measured in the analyzed hair tresses and the percentage variation in total proton content relative to their respective control tresses. Total proton content is indicative of the hair's hydration state. Indeed, water penetration into the hair results in an increase in total proton content. Compared with untreated control tresses, a slight increase in total proton content is observed for the placebo. A much more significant increase—around 12% compared with untreated controls—is observed following application of active A (PinoPlex), consistent with enhanced water penetration into the hair.




▲ **Figure 3a:** Total proton content of the different tresses.

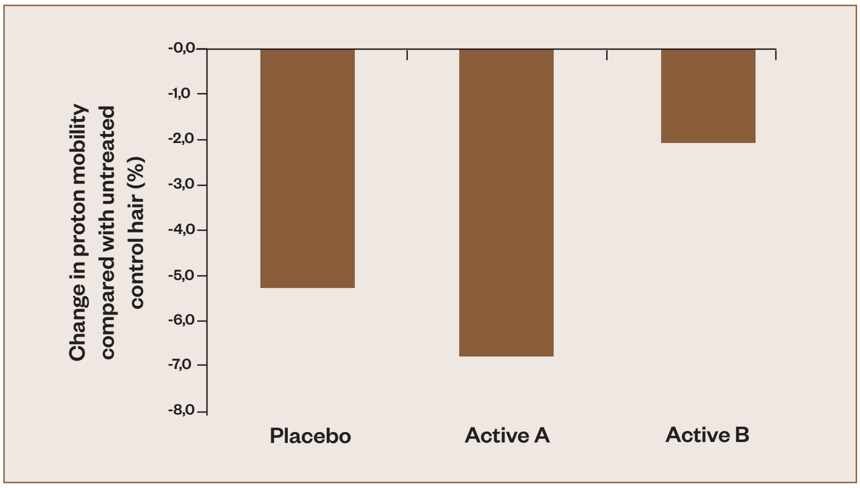



▲ **Figure 3b:** Percentage variation in total proton content compared with the control.



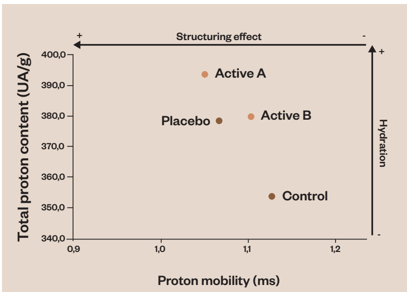

▲ **Figure 4a:** Proton mobility of the different tresses.

Conversely, application of active B shows no moisturizing efficacy compared with the placebo and can therefore be considered a negative control in this study.
Figures 4a and 4b show, respectively, the average proton mobility in the analyzed hair tresses and the percentage variation in proton mobility relative to their respective control tresses.
Proton mobility reflects the structural state of the hair and provides insight into the interaction forces between water, active molecules, and the hair fiber. Lower proton mobility indicates stronger binding interactions, and vice versa.
Compared with untreated controls, a slight decrease in proton mobility is observed for the placebo.
Application of active A (PinoPlex) leads to a marked decrease in proton mobility, indicating a restructuring effect of the active on the hair fiber.
Active B shows only minimal impact on proton mobility.
Two key NMR parameters should be considered:
• total proton content, reflecting moisturizing efficacy, as it is representative of the amount of water within the hair fiber ;
• proton mobility, reflecting water-binding interactions within the hair and the structural state of the hair fiber
Figure 5 presents these two key parameters for each analyzed tress.
The two actives exhibit markedly different effects on hair. Active B shows no effect in this test. In contrast, active A (PinoPlex) results in a more hydrated and more structured hair fiber, leading to smoother and shinier hair.
In summary, the results demonstrate both moisturizing and reparative efficacy of active A (PinoPlex) on hair fibers damaged by bleaching. Active B does not exhibit such efficacy. Figure 6 illustrates the demonstrated performance of active A.
Hair fibers sensitized by bleaching, due to damaged cuticles, have limited capacity to retain absorbed water (degradation of the keratin structure).



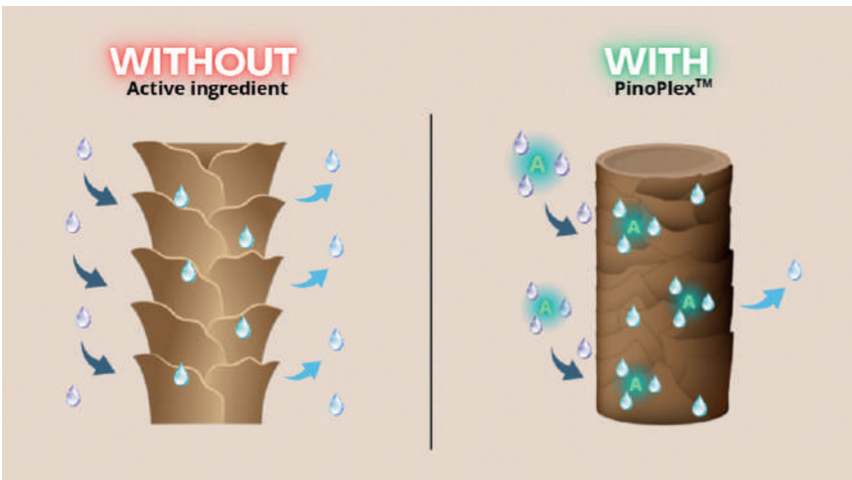

▲ **Figure 4b:** Proton mobility of the different tresses.

Depending on environmental humidity, water molecules can readily enter the fiber—but just as easily escape.
An active such as PinoPlex, owing to its molecular composition, has a strong ability to bind water in its environment. Upon penetrating the hair fiber, it significantly enhances hydration. Simultaneously, its penetration contributes to repairing the damaged hair structure, bringing it closer to that of a natural, non-sensitized fiber.




▲ **Figure 5:** Total proton content and proton mobility for the four hair tresses.

**Conclusion**
In conclusion, the methodologies developed by Cerevaa using NMR open up new application perspectives for measuring and monitoring hydration in keratin materials such as hair, skin, animal fur, and, more broadly, integumentary structures.
Understanding the efficacy of an active ingredient or formulation is thus made possible through the combined use of these methods with appropriate application protocols. ●




▲ **Figure 6:** Efficacy of PinoPlex active on hair.