RESOLUTION OENO 9/2002

MICROCRISTALLINE CELLULOSE

THE GENERAL ASSEMBLY.

HAVING CONSIDERED Article 5 of the October 13. 1954 International Convention on Unification of the Methods of Analysis and Appraisal of Wines.

WITH THE PROPOSAL of the Sub Commission on Methods of Analysis and Appraisal of Wines.

DECIDES to replace the existing monograph by the following monograph in the aforementioned International Oenology Codex:

MICROCRISTALLINE CELLULOSE

$(C_{12}H_{20}O_{10})$**n**

N° SIN : 460

1. OBJECT. ORIGIN AND FIELD OF APPLICATION

Microcrystalline cellulose is purified cellulose and is partially depolymerised. It comes from the treatment of alpha-cellulose mineral acids from plant fibres. Its molecular weight is approximately 36 000

Microcrystalline cellulose plays an important role in “supporting” very clarified fermentation as it increases the fermentability of the juices.

2. LABELLING

The concentration of the product must be mentioned on the label and if there is mixing as well as the method of preservation.

3. CHARACTERISTICS

Cellulose is found in microcrystalline powder form. white. odourless and tasteless. It is almost insoluble in water, acetone, ethanol, toluene, diluted acids and in 50 g/l sodium hydroxide solutions.

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4. IDENTIFICATION

4.1. In a watch glass.

Put approximately 10 mg of microcrystalline cellulose and add 2 ml of zinc chloride iodated solution (R). The solution turns bluish purple.

4.2. Degree of polymerisation

Put 1.300 g of microcrystalline cellulose in a 125 ml conical flask. Add 25 ml of water (R) and 25 ml of 1M cupriethylenediamine hydroxide. Immediately pass a nitrogen current. Close the flask and mix until completely dissolved. Pour 7 ml of the solution into an appropriate glass capillary viscosimetric tube. Time how long it takes between two lines on the viscosimeter and express the time measured in (t1). Calculate the kinematic viscosity V1 of the solution using the following formula:

$$V_1 = t_1(k_1)$$

In which k_1 is the viscosimeter constant.

Take out an appropriate volume of 1M cupriethylenediamine hydroxide and dilute with the same volume of water. (R). Using an appropriate capillary viscosimeter. determine the time flow of this solution.

Calculate the kinematics viscosity V_2 of the solvent using the following formula:

$$V_2 = t_2(k_2)$$

In which k_2 is the viscosimeter constant.

Determine the relative viscosity η_{rel} of the microcrystalline cellulose sample. using the following formula:

$$V_1/V_2$$

Determine the intrinsic viscosity $[\eta]c$ by extrapolation. using the intrinsic viscosity table in Annex.

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Calculate the degree of polymerisation P. using the formula:

$$P = 95\ [\eta]c/m\ [(100 - b)]/100$$

In which m is the mass. in grams of the trial and b is the value obtained in the test trial " loss through drying " in %.

The degree of polymerisation is not over 350.

4.3. pH

Shake for 20 minutes about 5 g of cellulose in 40 ml of water free of carbon dioxide. Centrifuge. The pH of the supernatant liquid must be between 5.0 and 7.5.

4.4. Soluble substances in ether

Prepare a column of 10.0 g of microcrystalline cellulose in a glass tube with an inside diameter of approximately 20 mm. Put 50 ml of ether free of peroxides (R). through the column and evaporate the eluate until bone dry. The residue should not be above 5.0 mg (0.05%).

4.5. Soluble substances in water

Mix 5.0 g of microcrystalline cellulose with 80 ml of water (R) for 10 mn. Filter in a vacuum and collect the filtrate in a weighed vase. Evaporate over a bath of 100° C water until bone dry and dry at 100-105°C for 1 hour. The residue is not above 12.5 mg (0.25%).

4.6. Starch

Add 90 ml of water (R) to 10 g microcrystalline cellulose. and boil for 5 mn. Filter when hot. Let cool and add 0.1 ml iodine 0.05 M to filtrate. There is no blue colouring.

4.7. Loss through drying

Put 1 g of cellulose in a mass capsule for 3 hours in an incubator at 100-105°C. Loss through drying should not be more than 6.0%.

All limits set below refer to the dried product.

4.8. Ashes

Incinerate at 600 ± 25°C the residue obtained in point 4.7. for 4 hours. The mass of the

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ashes should not be more than 0.1%.

4.9. Preparation of test solution

After weighing. dissolve the ashes in 2 ml of concentrated hydrochloric acid (R) and 10 ml of water (R). Heat to activate the dissolution and fill up to 50 ml with water.

4.10. Iron

Determine iron with an atomic absorption spectrophotometer following the method described in Chapter II into the test solution (4.9).

Iron content must be less than less or equal to 10 mg/kg.

4.11. Lead

Determine the lead according to the method described in Chapter II. into the test solution (4.9).

Lead content must be less than 5 mg/kg.

4.12. Mercury

Determine the mercury according to the method described in Chapter II

Mercury content must be less than 1 mg/kg.

4.13. Cadmium

Determine the cadmium according to the method described in Chapter II. into the test solution (4.9).

Cadmium content must be less than 1 mg/kg.

4.14. Arsenic

Determine the arsenic according to the method described in Chapter II.

Arsenic content must be less than 1 mg/kg.

4.15. Calcium

Determine the calcium with an atomic absorption spectrophotometer. following the method described in Chapter II. into the test solution (4.9).

Calcium content must be less than 500 mg/kg.

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5. STORING CONDITIONS

Cellulose must be stored in a well-ventilated place in sealed packages away from volatile substances which it might adsorb.

Declaration of Danemark

"When differences in specifications of purity, definitions and analytical methods exist between OIV and other competent intergovernmental organizations, such as Codex Alimentarius and European Union, Denmark believes that every possible effort must be done to identify why these differences exist and to reconcile them as far as possible, in order to avoid the existence of different international regulations on the same subject."

TABLE OF INSTRINSIC VISCOSITY

$$\frac{Intrinsic\ viscosity.\ [\eta]c.\ \ according\ to\ value\ of\ relative\ viscosity\ .\ \eta_{rel}}{[\eta]c}$$

η_{rel}	0.00	0.01	0.02	0.03	0.04	0.05	0.06	0.07	0.08	0.09
1,1	0,098	0,106	0,115	0,125	0,134	0,143	0,152	0,161	0,170	0,180
1,2	0,189	0,198	0,207	0,216	0,225	0,233	0,242	0,250	0,259	0,268
1,3	0,276	0,285	0,293	0,302	0,310	0,318	0,326	0,334	0,342	0,350
1.4	0,358	0,367	0,375	0,383	0,391	0,399	0,407	0,414	0,422	0,430
1,5	0,437	0,445	0,453	0,460	0,468	0,476	0,484	0,491	0,499	0.507
1,6	0,515	0,522	0,529	0,536	0,544	0,551	0,558	0,566	0,573	0,580
1,7	0,587	0,595	0,602	0,608	0,615	0,622	0,629	0,636	0,642	0,649
1,8	0,656	0,663	0,670	0,677	0,683	0,690	0,697	0,704	0,710	0,717
1.9	0,723	0,730	0,736	0,743	0,749	0,756	0,762	0,769	0,775	0,782

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2.0	0,788	0,795	0,802	0,809	0,815	0,821	0,827	0,833	0,840	0,846
2.1	0,852	0,858	0,864	0,870	0,876	0,882	0,888	0,894	0,900	0,906
2,2	0,912	0,918	0,924	0,929	0,935	0,94 1	0,948	0,953	0,959	0,965
2,3	0,971	0,976	0,983	0,988	0,994	1,000	1,006	1,011	1,017	1,022
2.4	1,028	1,033	1,039	1,044	1,050	1,056	1,061	1,067	1,072	1,078
2,5	1,083	1,089	1,094	1,100	1,105	1,111	1,116	1,121	1,126	1,131
2,6	1,137	1,142	1,147	1,153	1,158	1,163	1,169	1,174	1,179	1,184
2,7	1,190	1,195	1,200	1,205	1,210	1,215	1,220	1,225	1,230	1,235
2,8	1,240	1,245	1,250	1,255	1,260	1,265	1,270	1,275	1,280	1,285
2,9	1,290	1,295	1,300	1,305	1,310	1,314	1,319	1,324	1,329	1,333
3,0	1,338	1,343	1,348	1,352	1,357	1,362	1,367	1,371	1,376	1,381
3,1	1,386	1,390	1,395	1,400	1,405	1,409	1,414	1,418	1,423	1,427
3,2	1,432	1,436	1,441	1,446	1,450	1,455	1,459	1,464	1,468	1,473
3,3	1,477	1,482	1,486	1,491	1,496	1,500	1,504	1,508	1,513	1,517
3,4	1,521	1,525	1,529	1,533	1,537	1,542	1,546	1,550	1,554	1,558
3,5	1,562	1,566	1,570	1,575	1,579	1,583	1,587	1,591	1,595	1,600
3,6	1;604	1,608	1,612	1,617	1,621	1,625	1,629	1,633	1,637	1,642
3,7	1,646	1,650	1,654	1,658	1,662	1,666	1,671	1,675	1,679	1,683
3,8	1,687	1,691	1,695	1,700	1,704	1,708	1,712	1,715	1,719	1,723
3,9	1,727	1,731	1,735	1,739	1,742	1,746	1,750	1,754	1,758	1,762
4,0	1,765	1,769	1,773	1,777	1,781	1,785	1,789	1,792	1,796	1,800

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4,1	1,804	1,808	1,811	1,815	1,819	1,822	1,826	1,830	1,833	1,837
4,2	1,841	1,845	1,848	1,852	1,856	1,859	1,863	1,867	1,870	1,874
4,3	1,878	1,882	1,885	1,889	1,893	1,896	1,900	1,904	1,907	1,911
4,4	1,914	1,918	1,921	1,925	1,929	1,932	1,936	1,939	1,943	1,946
4,5	1,950	1,954	1.957	1,961	1,964	1,968	1,971	1,975	1,979	1,982
4,6	1,986	1,989	1,993	1,996	2,000	2,003	2,007	2,010	2,013	2,017
4,7	2,020	2,023	2,027	2,030	2,033	2,037	2,040	2,043	2,047	2,050
4,8	2,053	2,057	2,060	2,063	2,067	2,070	2,073	2,077	2,080	2,083
4,9	2,087	2,090	2,093	2,097	2,100	2,103	2,107	2,110	2,113	2,116

5,0	2,119	2,122	2,125	2,129	2,132	2,135	2,139	2,142	2,145	2,148
5,1	2,151	2,154	2,158	2,160	2,164	2,167	2,170	2,173	2,176	2,180
5,2	2,183	2,186	2,190	2,192	2,195	2,197	2,200	2,203	2,206	2,209
5,3	2,212	2,215	2,218	2,221	2,224	2,227	2,230	2,233	2,236	2,240
5,4	2,243	2,246	2,249	2,252	2,255	2,258	2,261	2,264	2,267	2,270
5,5	2,273	2,276	2,279	2,282	2,285	2,288	2,291	2,294	2,297	2,300
5,6	2,303	2,306	2,309	2,312	2,315	2,318	2,320	2,324	2,326	2,329
5,7	2,332	2,335	2,338	2,341	2,344	2,347	2,350	2,353	2,355	2,358
5,8	2,361	2,364	2,367	2,370	2,373	2,376	2,379	2,382	2,384	2,387
5,9	2,390	2,393	2,396	2,400	2,403	2,405	2,408	2,411	2,414	2,417

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6,0	2,419	2,422	2,425	2,428	2,431	2,433	2,436	2,439	2,442	2,444
6,1	2,447	2,450	2,453	2,456	2,458	2,461	2,464	2,467	2,470	2.472
6,2	2,475	2,478	2,481	2,483	2,486	2,489	2,492	2,494	2,497	2,500
6,3	2,503	2,505	2,508	2,511	2,513	2,516	2,518	2,52 1	2,524	2,526
6,4	2,529	2,532	2,534	2,537	2,540	2,542	2,545	2,547	2,550	2,553
6,5	2,555	2,558	2,561	2,563	2,566	2,568	2,571	2,574	2,576	2,579
6.6	2,581	2,584	2,587	2,590	2,592	2,595	2,597	2,600	2,603	2,605
6,7	2,608	2,610	2,613	2,615	2,618	2,620	2,623	2,625	2,627	2,630
6,8	2,633	2,635	2,637	2,640	2,643	2,645	2,648	2,650	2,653	2,655
6,9	2,658	2,660	2,663	2,665	2,668	2,670	2,673	2,675	2,678	2,680
7,0	2,683	2,685	2,687	2,690	2,693	2,695	2,698	2,700	2,702	2,705
7,1	2,707	2,710	2,712	2,714	2,717	2,719	2,721	2,724	2,726	2,729
7,2	2,731	2,733	2,736	2,738	2,740	2,743	2,745	2,748	2,750	2,752
7,3	2,755	2,757	2,760	2,762	2,764	2,767	2,769	2,771	2,774	2,776
7,4	2,779	2,781	2,783	2,786	2,788	2,790	2,793	2,795	2,798	2,800
7,5	2,802	2,805	2,807	2,809	2,812	2,814	2,816	2,819	2,821	2,823
7,6	2,826	2,828	2,830	2,833	2,835	2,837	2,840	2,842	2,844	2,847
7,7	2,849	2,851	2,854	2,856	2,858	2,860	2,863	2,865	2,868	2,870
7,8	2,873	2,875	2,877	2,879	2,881	2,884	2,887	2,889	2,891	2,893
7,9	2,895	2,898	2,900	2,902	2,905	2,907	2,909	2,911	2,913	2,915

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8,0	2,918	2,920	2,922	2,924	2,926	2,928	2,931	2,933	2,935	2,937
8,1	2,939	2,942	2,944	2,946	2,948	2,950	2,952	2,955	2,957	2,959
8,2	2,961	2,963	2,966	2,968	2,970	2,972	2,974	2,976	2,979	2,981
8,3	2,983	2,985	2,987	2,990	2,992	2,994	2,996	2,998	3,000	3,002
8,4	3,004	3,006	3,008	3,010	3,012	3,015	3,017	3,019	3,021	3,023
8,5	3,025	3,027	3,029	3,031	3,033	3,035	3,037	3,040	3,042	3,044
8,6	3,046	3,048	3,050	3,052	3,054	3,056	3,058	3,060	3,062	3,064
8,7	3,067	3,069	3,071	3,073	3,075	3,077	3,079	3,081	3,083	3,085
8,8	3,087	3,089	3,092	3,094	3,096	3,098	3,100	3,102	3,104	3,106
8,9	3,108	3,110	3,112	3,114	3,116	3,118	3,120	3,122	3,124	3,126

9.0	3,128	3,130	3,132	3,134	3,136	3,138	3,140	3,142	3,144	3,146
9.1	3,148	3.150	3,152	3,154	3,156	3,158	3,160	3.162	3,164	3,166
9,2	3,168	3,170	3,172	3,174	3,176	3,178	3,180	3,182	3,184	3,186
9,3	3,188	3,190	3,192	3,194	3,196	3,198	3,200	3,202	3,204	3,206
9,4	3,208	3,210	3,212	3,214	3,215	3,217	3,219	3,221	3,223	3,225
9,5	3,227	3,229	3,231	3,233	3,235	3,237	3,239	3,241	3,242	3,244
9,6	3,246	3,248	3,250	3,252	3,254	3,256	3,258	3,260	3,262	3,264
9,7	3,266	3,268	3,269	3,271	3,273	3,275	3,277	3,279	3,281	3,283

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9,8	3,285	3,287	3,289	3,291	3,293	3,295	3,297	3,298	3,300	3,302
9,9	3,304	3,305	3,307	3,309	3,311	3,313	3,316	3,318	3,320	3,321
10	3,32	3,34	3,36	3,37	3,39	3,41	3,43	3,45	3,46	3,48
11	3,50	3,52	3,53	3,55	3,56	3,58	3,60	3,61	3,63	3,64
12	3,66	3,68	3,69	3,71	3,72	3,74	3,76	3,77	3,79	3,80
13	3,80	3,83	3,85	3,86	3,88	3,89	3,90	3,92	3,93	3,95
14	3,96	3,97	3,99	4,00	4,02	4,03	4,04	4,06	4,07	4,09
15	4,10	4,11	4,13	4,14	4,15	4,17	4,18	4,19	4,20	4,22
16	4,23	4,24	4,25	4,27	4,28	4,29	4,30	4,31	4,33	4,34
17	4,35	4,36	4,37	4,38	4,39	4,41	4,42	4,43	4,44	4,45
18	4,46	4,47	4,48	4,49	4,50	4,52	4,53	4,54	4,55	4,56
19	4,57	4,58	4,59	4,60	4,61	4,62	4,63	4,64	4,65	4,66

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