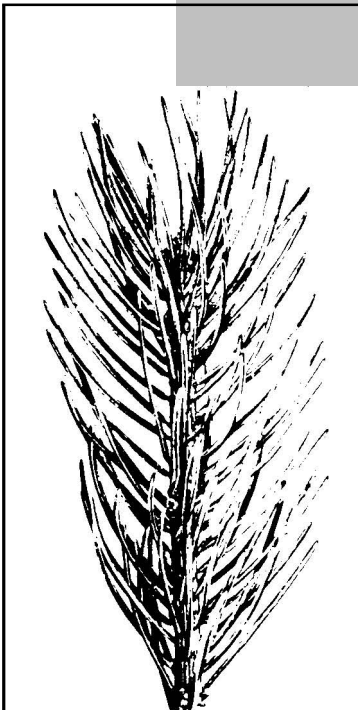


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**ENGELMANN SPRUCE, SUBALPINE FIR, AND LODGEPOLE PINE
MIXTURES FOR BLEACHED GROUNDWOOD, KRAFT, AND
SULFITE VISCOSE-GRADE PULP**



U. S. DEPARTMENT OF AGRICULTURE
FOREST SERVICE
FOREST PRODUCTS LABORATORY
MADISON WIS

SUMMARY

Three fairly abundant western softwoods were combined in mixture for pulping and bleaching experiments to determine their suitability for pulps of three types--groundwood, kraft, and viscose-grade sulfite.

A good-quality groundwood pulp, suitable for newsprint, production, was obtained from a mixture of 65 percent Engelmann spruce and equal parts of subalpine fir and lodgepole pine. The groundwood pulp responded well to peroxide and hydrosulfite bleaching.

The kraft pulp made from this same softwood mixture had a yield of 46.3 percent and a permanganate number of 19.5, and its strength was in the high range of kraft pulps. In bleaching, a brightness as high as 91 percent was obtained.

The viscose-grade sulfite pulp compared favorably in the usual test values with those published for commercial pulps. Its alpha-cellulose content was 95 percent, and the yield was high--40 percent.

ENGELMANN SPRUCE, SUBALPINE FIR, AND LODGEPOLE PINE MIXTURES FOR BLEACHED GROUNDWOOD, KRAFT, AND SULFITE VISCOSE-GRADE PULP¹

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INTRODUCTION

This study provides information on recent pulping and bleaching experiments with certain mixtures of three fairly abundant western species--Engelmann spruce, subalpine fir, and lodgepole pine--to determine their suitability for pulping.

Proportions of woods used in the study corresponded to the estimated availability in the area being considered for a pulping operation. Emphasis was placed on bleached and purified grades of

pulp.

From inquiries received at the Forest Products Laboratory, it is apparent that there is considerable interest in the possibilities for increased use of groundwood pulps by bleaching. Consideration was also given to the relative merits of five- and six-stage bleaching processes for kraft pulp, based on chlorine dioxide, as well as selecting types of pulp for production.

SPECIES USED

The species sampled were from the Pacific Northwest. They were identified at the Forest Products Laboratory as Engelmann spruce (*Picea engelmannii*), subalpine fir (*Abies lasiocarpa*), and lodgepole pine (*Pinus contorta*). The density and bark content of these samples are given in table 1. The measurements were made on 1-inch-thick disks cut from 10 randomly selected logs of each species. The bark content by volume is based on the difference in volume of the disks before and after removal of the bark. Volume is determined by water dis-

Table 1.--Density and bark content of three western softwoods

Species	Density ¹	Bark content	
		By weight ²	By volume
	Lb. per cu. ft.	Percent	Percent
Engelmann spruce	23.1	8.6	8.2
subalpine fir	20.5	11.8	10.4
Lodgepole pine	25.4	6.0	7.0

¹On a moisture-free weight and green volume basis.

²On a moisture-free weight basis.

¹Acknowledgment is made to Axel Hyttinen, J. F. Landrie, and E. L. Keller of the Forest Products Laboratory who assisted in this study.

²Maintained at Madison, Wis., in cooperation with the University of Wisconsin.

placement. In density, the fir and pine specimens were comparable to those of previous samples examined at the Laboratory. The density of the

Engelmann spruce was, however, somewhat higher than of previous samples but still in the range for this species.

GROUNDWOOD PULP

Pulping

A good-quality groundwood pulp was obtained from the mixture of 65 percent Engelmann spruce, 18 percent subalpine fir, and 17 percent lodgepole pine, by weight, at a favorable energy consumption (table 2). The strength properties of the pulp were essentially the same as those of groundwood pulps made at the Laboratory

Table 2.--Some results of groundwood pulping of a mixture of Engelmann spruce, subalpine fir, and lodgepole pine¹

Grinding data (run 1209) ²	
Pit temperature.....°F.	160
Pressure of wood on stone.....p.s.i.	20
Rate of grinding... tons moisture-free wood per sq. ft per 24 hr.	1.47
Energy consumed..... hp. -days per ton of moisture-free wood.	52
Pulp properties:	
Freeness (Canadian Standard)..... ml.:	120
Burst factor.....	12.4
Tear factor.....	47.7
Breaking length..... m.:	2,870
Density..... g. per cc.:	0.35
Screen analysis (Bauer-McNett):	
Retained on 28-mesh..... pct.:	14.0
Retained between 28- and 48-mesh..... pct.:	19.0
Retained between 48- and 100-mesh..... pct.:	20.8
Retained between 100- and 200-mesh..... pct.:	14.2
Passing 200-mesh..... pct.:	32.0
Brightness..... pct.:	61.5
Opacity of a handsheet weighing 52 g/sq. m..... pct.:	91.2

¹The mixture consisted of 65 percent Engelmann spruce, 18 percent subalpine fir, and 17 percent lodgepole pine, by weight.

²A 60-grit aluminum oxide abrasive pulpstone with a well-conditioned but fairly sharp surface was used. Grinder pit consistency was 4 percent, peripheral speed 3,120 feet per minute, and stone immersion 9 inches.

previously from each of the individual species. It is also comparable to commercial softwood-groundwood pulps of newsprint grade. The brightness was 61.5 percent and the opacity was 91.2 percent.

Energy consumption was relatively low-- 52 horsepower-days per ton of moisture-free wood-- which corresponds to 56.5 horsepower-days of total energy per ton of air-dry pulp, assuming 93 percent yield and 90 percent motor efficiency.

Bleaching

Although the brightness of 61.5 percent of the groundwood pulp was adequate for newsprint, it was of interest to know if it could be bleached readily to the level required for groundwood magazine papers. To determine this, two 1-stage and one 2-stage bleaching trials were made using 1 percent hydrogen peroxide, 1 percent sodium hydrosulfite, and 1 percent of each applied under normal commercial conditions. The brightness gains in points were: 6.7, 11.8, and 15. Thus, the experimental pulp responded well to the bleaching, and could be used in the higher-grade printing papers.

KRAFT PULP

Chip Preparation

The pulpwood logs were handpeeled and converted into approximately 5/8-inch chips in a commercial-sized chipper. The chips were passed over a gyrating screen equipped with 1-1/4- and 1/4-inch mesh screens to remove the over- and under-sized materials. After the moisture content of the chips from each species was determined, the chips were mixed in the proportion of 65 percent Engelmann spruce, 18 percent subalpine fir, and 17 percent lodgepole pine, by weight. The average moisture content of the chip mixture was 32 percent.

Pulping and Results

After small-scale pulping trials to establish suitable cooking conditions, a digestion was made in a 14-cubic-foot stainless steel, steam-jacked, rotating digester to provide sufficient pulp for bleaching experiments. The conditions used were: a liquor-to-wood ratio of 4 to 1; 25.5 percent sulfidity (based on active alkali); 22.5 percent NaOH + Na₂S (moisture-free wood basis); 1.5 hours to 170° C.; and 1.5 hours at 170°C. (table 2).

The internal digester pressure was lowered

to about 80 p.s.i.g., and then the digester charge was "blown" into a stainless steel blow tank where the pulp was washed with a cold water shower. The washed pulp was screened through a 0.012-inch slotted flat screen and dewatered on a wet machine. Later, the pulp was cleaned centrifugally.

As shown in table 3, the pulp prepared in the pilot plant equipment had a screened yield of 46.3 percent with 0.1 percent screenings. The permanganate number was 19.5, when using 25 milliliters of 0.1N potassium permanganate according to TAPPI Standard T 214.

Table 3.--Conditions and some results of kraft pulping of mixed western softwoods¹

Digestion No.....	4352
Chemicals charged:	
Initial concentration calculated as Na ₂ O .g. per l.:	43.8
NaOH + Na ₂ S.....	72.5
Sulfidity (based on active alkali).....	25.5
Liquor-to-wood ratio.....	4:1
Time to 170° C.....	1.5
Time at 170° C.....	1.5
Total chemicals consumed ²	14.9
Yield: ²	
Screened.....	46.3
Screenings.....	0.1
Lignin ³	5.4
Permanganate number.....	19.5
Disperse viscosity ⁴	32.6

¹Mixture consisted of 65 percent of Engelmann spruce, 18 percent of subalpine fir, and 17 percent of lodgepole pine.

²On a moisture-free wood basis.

³TAPPI Standard T 222.

⁴Pulp at 0.5 percent concentration in cupriethylenediamine. TAPPI Standard T 230.

Bleaching

Samples of the kraft pulp were bleached to a brightness of 88 to 91 percent by five- and six-stage processes with total chlorine equivalents of both 10.3 and 11.0 percent in each process (table 4). The results obtained with these two processes are summarized in table 5.

The higher chlorine equivalent (11 per cent)

developed about 2 points higher brightness in each process. The six-stage process consisted of chlorination, extraction, hypochlorite, chlorine dioxide, extraction, and chlorine dioxide. One 5-stage process was the same as the six-stage process except the hypochlorite stage was not used. The conventional five- and six-stage processes at equal chlorine equivalent compared as follows:

(1) The six-stage process required 5 to 5.5 hours longer reaction time at maximum brightness, developed 1.5 to 1.7 points higher brightness, but showed no difference in brightness stability nor in strength of bleached pulp.

(2) The yield of bleached pulp was possibly a little lower with the six-stage process.

(3) At equal brightness, the reaction time for the six-stage process was only 2-1/4 hours longer than the five-stage process.

The other five-stage process (bleach 4950-II) included first- and second-stage oxidative techniques, or prehypochlorite and oxidative extraction stages. In applying prehypochlorite, the chlorine is added immediately after exhaustion of the hypochlorite without intermediate washing. In this instance, brightness was improved considerably.

The purpose of oxidative bleaching is to apply the tolerable maximum of hypochlorite under conditions that minimize degradation of the cellulose as it relates to pulp strength. It is especially useful for three-stage bleaching of hardwood kraft pulps.³ In more recent work at the Laboratory, eucalyptus kraft pulps ranging in Kappa number from 20 to 40 were bleached to a brightness of 84 to 86 without loss in strength. Three stages--prehypochlorite, oxidative extraction, and sodium hypochlorite--were used. Total chlorine equivalents ranged from 6 to 10 percent.

Commercial applications of the prehypochlorite stage, exclusive of oxidative extraction, have been reported.^{4,5}

Strength

Strength development data for the kraft pulp, before and after bleaching, are given in table 6.

³Simmonds, F. A., and Keller, E. L. Bleached kraft pulp from several southern hardwoods. southern Pulp and Paper Manufacturer 26(8): 57-58, 60, 62 68. 1963.

⁴Evans, John C. W. Brown's new bleach plant has many interesting points. Paper Trade Jour. 141(7): 44-49. 1957

⁵Ferguson, J. M., and MacDonald, E. A. Bleaching of alkaline hardwood pulp at Cornwall, Ontario. Paper Trade Jour. 143(3): 42-46. 1959

Table 4--Some results of bleaching a mixed western softwood kraft pulp by three processes¹

Bleach No.	Stage No.	Bleaching chemicals ²		Temperature	Consistence	Duration	pH-ion concentration		Yield	Brightness		Dispersive viscosity ³	
		Name	Amount applied				Amount consumed	Initial		Final	Basis unbleached pulp		Basis wood
		Percent	Percent	°C.	Percent	Min.	pH	pH	Percent	Percent	Percent	Centipoises	
4946	1	Chlorine	7.3	6.9	25	2.0	60	2.4	2.2				
	2	Sodium hydroxide	3.0		70	10.0	60	11.8	11.5				
	3	Chlorine dioxide	2.0	1.9	70	8.0	60	(5)	4.2				
	4	Sodium hydroxide	1.0		50	8.0	60	12.1	11.7				
	5	Chlorine dioxide	1.0	1.0	70	8.0	195	(5)	4.2		87.8	21.0	
Total available chlorine		10.3	9.8										
4950 I	1	Chlorine	7.6	6.9	25	2.0	90	2.1	1.8				
	2	Sodium hydroxide	3.0		70	10.0	60	11.8	11.5				
	3	Chlorine dioxide	2.2	2.1	70	8.0	150	(5)	4.2				
	4	Sodium hydroxide	1.0		50	8.0	60	11.7	11.5				
	5	Chlorine dioxide	1.2	1.0	70	8.0	240	(2)	4.2	94	43.5	89.6	84.5
Total available chlorine		11.0	10.0										
4950 II	1	Calcium hypochlorite	3.8	3.8	25	8.0	30	11.3	8.8				
		Chlorine	3.8	3.4	25	3.0	60	2.6	2.2				
	2	Calcium hypochlorite	1.0	1.0	45	10.0	60	12.0	11.6				
		Sodium hydroxide	3.0										
	3	Chlorine dioxide	1.6	1.2	70	10.0	120	6.0	3.4		78.6	19.2	
4	Sodium hydroxide	.5		50	10.0	60	11.5	11.4					
5	Chlorine dioxide	.8	.8	70	10.0	240	6.0	4.3		90.0	87.5	18.1	
Total available chlorine		11.0	10.2										
4947	1	Chlorine	7.3	6.88	25	2.0	60	2.4	2.2				
	2	Sodium hydroxide	3.0		70	10.0	60	11.8	11.5				
	3	Sodium hypochlorite	1.0	1.0	37	8.0	100	10.9	10.9				
	4	Chlorine dioxide	1.58	1.48	70	8.0	210	(5)	3.6				
	5	Sodium hydroxide	.5		50	8.0	60	11.8	11.5				
	6	Chlorine dioxide	.53	.50	70	8.0	240	(5)	4.1		89.3	19.4	
Total available chlorine		10.41	9.86										
4951	1	Chlorine	7.6	6.92	25	2.0	90	2.1	1.9				
	2	Sodium hydroxide	3.0		70	10.0	60	11.7	11.5				
	3	Sodium hypochlorite	1.3	1.09	37	8.0	240	11.2	10.4				
	4	Chlorine dioxide	1.5	1.24	70	8.0	240	(5)	3.7				
	5	Sodium hydroxide	1.0		50	8.0	60	11.8	11.6				
	6	Chlorine dioxide	.6	.53	70	8.0	240	(5)	4.5	93.4	43.3	91.3	86.4
Total available chlorine		11.0	9.78										

¹Digestion No. 4352, 65 percent Engelmann spruce, 18 percent subalpine fir, 17 percent lodgepole pine. Permanganate number: 19.5. Lignin, 5.4 percent.

²Values are based on unbleached pulp (moisture-free) and those for chlorine compounds refer to available chlorine

³Pulp at 0.5 percent concentration in cupriethylenediamine. TAPPI Standard T 230.

⁴One hour at 105° C. in a forced-circulation oven

⁵pH of pulp-water mixture adjusted to 5.4 before addition of chlorine dioxide solution.

⁶Bleaches 4946 and 4947 were made with 50 grams of pulp each, and 4950 and 4951 with 908 gram.

Table 5.--Comparison of the five- and six-stage bleaching processes

Number of stages	Total chlorine applied	Total reaction time	Increase in time	Brightness	Increase in brightness	Loss in brightness after aging ¹	Yield of pulp	Strength of bleached pulp
Percent	Hr.	Min.	Hr.	Percent	Percentage points	Percentage points	Percent	
5	10.3	7	15	87.8				
6	10.4	12	17	89.3	1.5			
5	11.0	10	0	(2)	89.6	5	94.0	(3)
5	11.0	9	30	2.25	90.0	.4	2.5	
6	11.0	15	30	5.5	91.3	1.7	5	(3)

¹Heated for 1 hour at 105° C. in a forced-circulation oven

²At equal brightness, the six-stage process required only 2-1/4 hours longer reaction time.

³In the freeness range of 350 to 250 milliliters, Canadian Standard, the bleached pulps were essentially equal in strength and also to the unbleached pulp except in tearing resistance, which averaged 10 percent lower (table 6).

Table 6.--Strength development of mixed western softwood kraft pulp before bleaching, and after bleaching by two processes¹

Pulp	: Beating time	: Freeness (Canadian Standard)	: Burst factor	: Tear factor	: Folding endurance (M.I.T.)	: Breaking length	: Density	: Disperse viscosity
	: Min.	: Ml.			: Double folds	: M.	: G. per cc.	: Centipoises
Unbleached (Digestion No. 4352)	0	690	40	264	335	6,700	0.46	32.6
	32	550	96	153	1,300	12,300	.66
	45	450	102	140	1,900	13,400	.72
	57	350	105	124	2,250	14,300	.75
Bleach No. 4950 (5 stages)	0	685	35	319	222	5,020	.46	19.4
	20	550	105	135	1,727	11,700	.71
	32	450	106	118	1,800	13,200	.73
	42	350	107	111	2,300	14,000	.76
Bleach No. 4951 (6 stages)	0	680	37	293	127	4,850	.48	13.9
	20	550	96	128	1,760	12,140	.70
	29	450	100	117	2,000	12,800	.73
	38	350	102	110	2,200	13,000	.76

¹TAPPI Standards T 200 and T 205. The test values were interpolated from curves, except those for the unbeaten pulps.

The strength of the unbleached pulp was in the high range, and typical of kraft pulps from similar species. In tearing resistance, it was somewhat lower than often found for Douglas-fir kraft pulps.

Upon bleaching, the tearing resistance of the bleached pulps in the unbeaten condition was increased 20 percent by the five-stage process and 11 percent by the six-stage process. However, after being beaten to a Canadian Standard freeness of 350 the bleached pulps were 11 percent lower in tear factor. Other strength properties were not decreased by the bleaching.

Because of the relatively high viscosity values of the bleached pulps (19.4 and 13.9 centipoises),

which were known in advance of the strength evaluation, the loss in tearing strength upon beating was unexpected.

It is commonly accepted that the strength of kraft pulps is related to their degree of polymerization (DP), and that an index of this is the disperse viscosity of the pulp in a carbohydrate solvent such as cupriethylenediamine.

If the DP is not decreased below 1,000 during bleaching, usually the pulp strength is not seriously affected.^{6,7} (A DP of 1,000 corresponds to a viscosity of 10 centipoises for a 0.5 percent concentration of pulp in cupriethylenediamine.) The paper by Dahm⁷ reviews literature of this subject. He concluded that when pulp is degraded

⁶Casciani, F., and Storin, G. K. Chlorine dioxide and alkaline peroxide in kraft bleaching. Tappi 33(12): 588-594. 1950.

⁷Dahm, H. P. On the relation between pulp viscosity and paper strength of sulfate pulp degraded by chlorine-hypochlorite bleaching. (In English) Norsk Skogindustri 13(6): 188-192. 1959.

by bleaching, the sheet strength is predominantly dependent on fiber strength at a threshold DP of about 1,150 for beaten pulp. Above this level, fiber-to-fiber bonding strength is of considerable influence.

The pulp from the oxidative bleaching (4950 II) was not tested for strength. In other work, however, a kraft pulp made from a mixture of

western woods which included 9 percent of Engelmann spruce and 6 percent of subalpine fir was bleached with this technique in 4 stages to a brightness of 86 percent without loss in strength. Hydrogen peroxide was applied in the fourth stage. The permanganate number of the pulp was 18.6, and the total chlorine equivalent was 10 percent.

SULFITE PULP

Preparation

After orienting trials were made, a calcium base sulfite pulp was prepared from a wood mixture consisting 78 percent of Engelmann spruce and 22 percent of subalpine fir. Pulping conditions are given in table 7, as are yield and lert values. The values show that the unbleached

Table 7.--Conditions and some results of the sulfite pulping of a mixture of 78 percent of Engelmann spruce and 22 percent of subalpine fir.

Item	Result
Liquor charged:	
Base.....	Calcium
Total sulfur dioxide.....pct.:	7.4
Combined sulfur dioxide.....pct.:	.97
Amount charged per 100 pounds of moisture-free wood.....gal.:	¹ 53.2
Temperature schedule:	
Time to 80° C.....hr.:	.5
Time from 80° to 115° C.....hr.:	1.75
Time at 115° C.....hr.:	1.0
Time from 115° to 150° C.....hr.:	1.5
Time at 150° C.....hr.:	1.5
Pressure.....lb. per sq. in.:	90
Yield: ²	
Screened.....pct.:	46.0
Screenings.....pct.:	.65
Permanganate number.....	8.0
Alpha-cellulose ³pct.:	88.0
Pentosans.....pct.:	3.0
Mannans.....pct.:	6.0
Disperse viscosity ⁴centipoises:	26.4

¹Including the moisture in the chips, this was equivalent to 60 gallons of liquor in the chips.

²On a moisture-free wood basis.

³Not corrected for lignin.

⁴Pulp at 0.5 percent concentration in cupriethylenediamine. TAPPI Standard T 4520

pulp was well within the range of the quality required for this type of pulp.

Purification

The pulp was purified in four stages, consisting of chlorination, extraction with caustic soda at 107° C., sodium hypochlorite and chlorine dioxide (table 8). The purified pulp was steeped in hydrochloric acid solution to lower the iron content, commonly found to be high in laboratory pulps.

Table 8.--Purification of sulfite pulp ¹

Stage No.	Chemical	Amounts ²	Consistence	Temperature	pH value	Duration
		Applied:	Con- sumed:		Initial:	Final:
		Percent:	Per- cent:	°C.		Min.
1	:Chlorine	: 2.3	: 2.2	: 2	: 24	: 2.1 : 2.1 : 90
2	:Sodium : hydroxide	: 7.34	:	: 7	: 107	: 12.5 : 12.5 : 60
3	:Sodium : hypochlorite:	: .3	: .26	: 10	: 40	: 10.9 : 9.4 : 240
4	:Chlorine : dioxide	: .4	: .32	: 10	: 70	: 6.3 : 6.4 : 210
³ 5	:Hydrochloric : acid	:	:	: 4	: 23	: 2.0 : 2.0 : 120

¹Pulp consisted of 78 percent Engelmann spruce and 22 percent subalpine fir.

²Percentages are based on weight of unbleached pulp, moisture-free, and those for chlorine compounds refer to available chlorine equivalents.

³To lower the high content commonly found in laboratory experimental pulp production.

Results of tests made on the purified pulp were:

Yield:
Pulp basis 87.1 pct.
Wood basis 40.1 pct.

Brightness, G. E. equivalent	93.9	pct.
Alpha-cellulose	95.1	pct.
Beta-cellulose	.9	pct.
Gamma-cellulose	3.9	pct.
Pentosans	2.2	pct.
Solubility in:		
7.14 percent caustic soda	10.4	pct.
Ether	.11	pct.
Ash	.05	pct.
Disperse viscosity [§]	25.8	centipoises
Mannans	1.9	pct.

The 40 percent yield, at an alpha-cellulose content of 95 percent, was excellent. The beta-cellulose, pentosan, and mannan values were favorably low. Although the solubility in hot caustic soda was at the tolerable maximum, this could be improved by minor variations during preparation. The disperse viscosity was in the high viscosity tire cord grade of softwood sulfite pulp.

Thus, the experimental pulp was in the quality range of commercial viscose grade of pulps.

CONCLUSION

Good-quality bleached groundwood and kraft pulps and sulfite viscose-grade pulps can be produced readily from Engelmann spruce, sub-alpine fir, and lodgepole pine, with exclusion of the pine from the mixture for the viscose pulp.

The technique of first- and second-stage oxidative bleaching--prehypochlorite and oxidative extraction--is worthy of consideration for bleaching kraft pulps in general to a moderate brightness level in three stages or, in the case of hardwood kraft pulps, to a high level.

[§]Pulp at 0.5 percent concentration in cupriethylenediamine. TAPPI Standard T 230. At 1 percent concentration the viscosity was 223 centipoises.

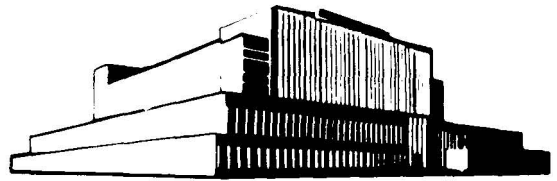
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