sawdust offers a potential saving of 23%over manure, if material costs, overhead, and yields are the same. Where the sawdust would be available at no cost, the saving would be 55%.

As shown in the photographs, P. ostreatus is very different from A. campestris. Pleurotus does not grow in the familiar button form characteristic of young Agaricus. It does not have the thick "meaty" tissue of the Agaricus buttens. In shape and growth it resembles more closely the Japanese shiitake mushroom. Like the latter, it has a tough, inedible stem. The flavor, however, is more like that of A. campestris. Pleurotus would appear to have its greatest value as a flavoring ingredient for soups, sauces, and gravies rather than as a fresh mushroom for table use

Mushrooms are a source of essential amino acids and of the B vitamins (1, 4, 7, 8). They can be "factory-produced" the year around from waste products which originate from non-

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depleting sources and may be visualized as an important food source for the future. Full realization of the market potential, however, awaits more efficient production and lower cost.

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# **Morphine Extraction from Domestically Grown Opium Poppy**

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To meet the needs of our nation in an emergency, a process was developed to recover crude morphine liquor from mature, domestically grown, opium poppy plants. This liquor can be used by licensed refiners as a substitute for opium to produce medicinal morphine, if U.S. opium imports should be severely limited or cut off. Over 95% of the morphine was extracted from meal with water-saturated isobutyl alcohol in a continuous countercurrent extractor. In preparing the meal, stems and seed were separated from capsular material by a four-step size-reduction procedure with a 6% loss of morphine Morphine concentration in the meal was increased from 0.5 to 0.7%. An aqueous ammonia treatment of the meal prior to extraction liberated the alkaloids from their naturally occurring salts. Poppy meal or straw keeps in dry storage without any morphine loss.

MPORTED OPIUM is the source of medicinal morphine in the United States. To ensure adequate supplies of morphine in a national emergency, work was undertaken to develop a method for recovering crule morphine liquor from the mature capsules of opium poppy plants grown in the United States.

Opium is the dried juice or latex of unripe capsules of a particular poppy species, Papaver somniferum. Although the opium poppy has been grown for thousands of years, opium is still obtained through much tedious manual work, both in cultivating the plant and in collecting the latex (2). Some 22

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alkalcids are found in opium, but only three-morphine, codeine, and papaverine-are of commercial importance. The amount of morphine varies over wide limits, but in a good grade of opium the content averages 10%, and the codeine and papaverine are present in much smaller amounts.

Interest in cheaper production methods led to the development of more modern processes for recovering opium alkaloids directly from the ripened poppy plant, and some of these processes were put into commercial operation on a very limited scale in Germany, Switzerland, and Hungary in the 1930's (5, 6, 9). From information given in the literature for these and other processes (1), each appears to have disadvantages.

Morphine Analyses. All of the samples were analyzed by the Matchett and Levine method (11, 16), but for a rapid estimate, a modification of Hanes' ferricyanide micromethod for determining reducing sugars (4) was employed.

Most analysts believe the Matchett and Levine method is insufficiently specific for morphine (16); therefore, a commercial morphine refiner checked duplicate samples from one of the extraction tests. The extract liquor was examined for its morphine content by a modified U. S. Pharmacopoeia method and a special method was used for the poppy meal (8).

Preparation of Poppy Meal. The opium poppy was grown under irrigation by the Field Crops Research Branch of the U. S. Department of Agriculture. At maturity the crop was harvested with a conventional small grain combine modified to produce three fractions: poppy seed, stems, and leaves which were returned to the harvest field, and crude straw—a mixture of capsules, stems, and seed (15).

The highest concentration of morphine is in the capsule of the plant, a little is in the stems, but none is in the seed (7). Much less material needs processing if stems are separated from the capsules; because straw must be protected from rain, such a separation reduces the investment for storage facilities. Seeds also should be removed, because their oil complicates morphine purification.

Preliminary milling studies indicated that a corrugated roller mill would produce flattened, partially cut stems and broken capsular material that could be separated by screening. Because capsular material could not be rolled and flaked satisfactorily prior to solvent extraction, as in oilseeds processing, it was ground into a meal passing a 20mesh sieve to facilitate extraction. Subsequently, the 4-break milling procedure illustrated in Figure 1 was developed and used to process a 4275pound lot of crude straw. Details of the milling conditions are given in Table I.

An Allis-Chalmers experimental roller mill equipped with LePage corrugated rolls measuring  $6 \times 6$  inches was employed for the first break and a 3-pair-high Robinson roller mill with  $9 \times 18$  inch conventional corrugated rolls was used for the next three breaks. Normally only one pair of rolls with proper corrugations and speed differentials is used for each break. Because of equipment limitations, two pairs were used for the third break and three pairs for the fourth. Any remaining oversize stock was ground in a No. 000 Abbe mill operated at 700 r.p.m. The Robinson rolls were operated sharp-to-sharp, but the cutting edges were dull. The milled stream from each break was screened in a Robinson grain cleaner, size 2U, operated without aspiration and equipped with screens having round-hole perforations of the sizes shown in Figure 1.

The resultant poppy meal constituted 69% of the crude straw, occupied only about one third the original volume, and had a bulk density of about 13 pounds per cubic foot. The morphine lost in all discarded stems totaled only 5.7% of that present in the crude straw. Removing these stems decreased the weight of the meal to be extracted by 22% and seed removal decreased it another 6.5%. In this way the morphine concentration in the meal was increased from approximately 0.5 to 0.7%.

If kept dry, the straw or meal can be stored throughout the year without loss of morphine. Meal stored in both

### Table I. Milling Conditions Used for Poppy Meal, Lot MS-7

Break	Type of Corrugated Roll	Clearance, Inch	Fast Roll, R.P.M.	Slow Roll, R.P.M.
1	LePage corrugations, 5 per inch	0.025	380	342
2	Conventional, spiral cut corrugations Top pair, 8 per inch	0.018	460	180
3	Conventional, spiral cut corrugations			
	Top pair, 8 per inch	0.010	460	180
	Middle pair, 12 per inch	0.008	511	<b>2</b> 01
4	Conventional, spiral cut corrugations			
	Top pair, 8 per inch	0.008	460	180
	Middle pair, 12 per inch	0.005	511	201
	Lower pair, 20 per inch	0.002	575	225



# Table II. Typical Conditions and Values for Extraction of Poppy Meal

Meal (air-dry) feed rate, lb./hr. Wetting liquor, rate, lb./hr.	12 20
Solvent A rate, lb./hr.	54.0
Solvent B rate, lb./hr.	26.2
Over-all solvent ratio	6.7
Solids retention time within	
extractor, min.	45
Extraction temperature, ° F.	140
Solvent feed temperatures, ° F.	135-140
Fines removed from extract	
liquor, lb./100 lb. poppy	
meal	1.75
Anhydrous ammonia rate, lb./	
100 lb. poppy meal	1.25
Morphine concentration in	
extract liquor, %	0.225

Figure 1. Diagrammatic flowsheet for milling poppy straw

Figure 2. Diagrammatic flowsheet for extraction and pressing operations POPPY NEAL ADUTOUS ANMONIA SOLVENT B SOLV

open and sealed containers kept at room temperature and in an unheated barn showed no loss of morphine in 2 years.

**Extraction of Alkaloids.** Morphine concentration in poppy meal is very low compared to the solute level in such materials as oilseeds which ordinarily are extracted in large-scale continuous extractors. However, it had been found previously that a high percentage of the morphine could be extracted in 1/2 hour (12, 13, 14); this is well within a practical range for continuous extraction.

Poppy meal wetted with extract liquor was fed through a gravimetric feeder followed by a screw conveyor and into a continuous countercurrent horizontal extractor of the Kennedy type (10) with 20 extraction stages. Poppy meal was introduced at stage 1, and two solvent streams were used, one entering at stage 20 and the other between stages 16 and 17. The extracted solids were drained for approximately 7 minutes on an inclined flight conveyor. Extract liquor was filtered through cloth bags made of cotton duck, style 8, to remove suspended solid particles. The extraction equipment was operated on a continuous basis for periods of 20 to 24 hours.

About 1 hour before the poppy meal was fed into the extractor, 6-pound batches were mixed with 10 pounds of fresh extract liquor to which 150 ml. of aqueous ammonia  $(25.3\% \text{ NH}_3)$  had been added. This treatment reduced the quantity of fines carried in the extract liquor and the ammonia liberated alkaloids from their naturally occurring salts. A diagrammatic flowsheet for the extraction and pressing operations is shown in Figure 2.

Typical flow rates and process conditions established after a number of exploratory runs are given in Tables II and III.

The resultant extract liquor contained 96.9% of the morphine in the poppy meal feed. Increasing the amount of wetting liquor decreased the yield of morphine somewhat and also increased

the quantity of suspended solids in the extract liquor. Thebaine and codeine were also extracted to about the same extent as morphine.

The change in morphine concentration of both solids and liquor as they pass through the extractor has considerable bearing on the number of extraction stages required. Data obtained from one of the earlier tests are shown in Table IV. Although the morphine contents for solids removed from the various extractor stages are not necessarily absolute values, they are indicative of the concentration gradient and of the rate at which morphine was extracted. Twenty stages were required in this instance to extract 90.3% of the morphine, based on the quantity in the extract liquor.

The primary solvent was a watersaturated solution of isobutyl alcohol. This solvent, A, entering at stage 20, was made up of alcohol recovered from desolventizing the meal and either anion effluent or redistilled isobutyl alcohol, depending upon whether alkaloids in the extract liquor had been recovered by the ion exchange or the distillation recovery method (3). When anion effluent was included, solvent A had a morphine concentration of 0.002%, and when redistilled alcohol was used the concentration naturally was zero. The secondary solvent, B, contained about 0.009% morphine and was the isobutyl alcohol liquor recovered by pressing the extracted meal.

Poppy meal was retained within the extractor for  $\frac{3}{4}$  hour, which is believed best for the conditions used. The optimum holding time was not determined.

Fines in the extract liquor averaged 1.87 pounds (air-dry solids) per 100 pounds of poppy meal fed to the extractor. No difficulty was encountered in the filtration step although more than one half of the fines passed a 100-mesh sieve and about 30% went through a 325-mesh sieve.

No attempt was made to study the influence of the other variables such as extraction temperature, quantity of ammonia used to treat the poppy meal, and fineness of grind for the meal.

Pressing Extracted Meal. The extracted meal was pressed in a Davenport continuous dewatering press, size 3A, equipped with revolving disks fitted with brass screens having 3/64-inch perforations and operated at its lowest speed, 0.51 r.p.m. Wet marc entering the press contained about 80% moisture and volatile matter; at an input rate of 2800 pounds per hour, the resultant pressed meal averaged 60.3% moisture and volatile matter. This is a reduction in total volatiles from 4.0 to 1.5 pounds per pound of dry solids. At an input rate of 6100 pounds per hour, there was a marked reduction in the quantity of press liquor produced.

## Table III. Material Balance for Extraction of Poppy Meal

	Morphine, Ounces/Hr.	Solids, Lb./Hr.	Total Stream, Lb./Hr.
Material in			
Solvent A	0.018	0,22	54.0
Solvent B	0.036	0.23	26.2
Wetted meal	2.065ª	11.31	32.0
Total	2.119	11.76	112.2
Material out			
Extracted meal	0.118*	11.05	53.9
Extract liquor	1.265	0.72	35.1
Wetting liquor	0.721	0.41	20.0
Fines and entrained liquor	0.0670	0.34	1.3
Total	2.171	12.52	110.3
Material in – material out	+0.052	+0.76	-1.9
Difference	+2.5%	+6.5%	-1.7%

 $^a$  12.0 lb. poppy meal, 0.70% morphine content, and 20.0 lb. wetting liquor containing 0.225% morphine.  $^b$  Assuming meal, dry-basis, contains 5% of the morphine in the dry feed solids and the entrained liquor contains 0.008% morphine.  $^\circ$  Assuming dry solids contain 0.6% morphine and entrained liquor contains 0.225% morphine.

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### Table IV. Typical Extraction Profile

	Concentration, %		
	Solidsa	Liquor <sup>b</sup>	
Poppy meal feed, at $8.6\%$			
moisture	0.70		
Liquor leaving stage 0		0.221	
Solids leaving stage 1	0.46		
Material leaving stage 2	0.34	0.178	
Material leaving stage 4	0.26	0.135	
Material leaving stage 6	0.20	0.092	
Material leaving stage 8	0.16	0.070	
Material leaving stage 14	0.09	0.025	
Material leaving stage 20	0.04	0.008	
Solvent A, entering stage			
20		0,002°	
Solvent B, entering stage			
16/17		0.012	

<sup>a</sup> To minimize deposition of any morphine present in entrained extraction liquor, each sample when collected was wrung in a towel to remove excess liquor, washed once with pentane-hexane solvent in which morphine is insoluble, and then dried at 140° F. <sup>b</sup> Samples were filtered to remove suspended solids. <sup>c</sup> Ferricyanide method of analysis used.

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