



Photographic gelatin or emulsion purification (Larry D Hagemaiier, Barry M Brown, John C McFall, Elbert L Ray)

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## **Photographic gelatin or emulsion purification 13122**

This disclosure relates to photographic gelatin or emulsion washing procedures, and especially to methods for removing water soluble byproducts of the silver halide formation, from the peptized silver halide suspension of a photographic silver halide emulsion.

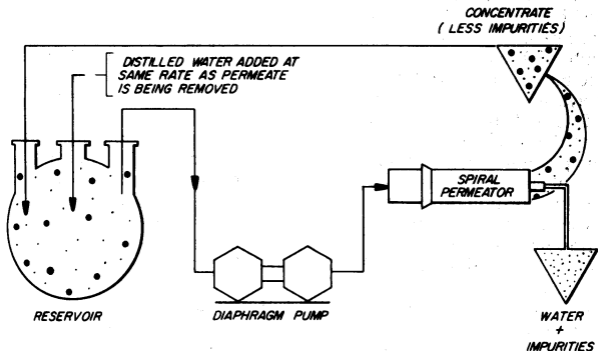
This disclosed method is particularly useful in removing soluble salts, especially nitrates, from photographic emulsions. The washing process does not result in significant silver or peptizer loss and has the advantage of low water usage and no cooling requirements.

Still other advantages include elimination of the need for special gelatin derivative or the need for special precipitants or coagulants or a need for a peptizing material able to gel upon cooling. The process is also readily adaptable to continuous operation.

The use of the spiral wound permeator configuration has the advantage of a high membrane working surface to permeator volume ratio not obtainable with tubular membrane configurations. In addition, the large feed flow channels allow processing of the colloidal silver suspension without the fouling and fiber breaking encountered with hollow fiber systems.

The above objects have been accomplished by employing the

# CONTINUOUS DIAFILTRATION



- - HIGH MOLECULAR WEIGHT PRODUCT (EMULSION)
- - LOW MOLECULAR WEIGHT IMPURITIES

continuous diafiltration system illustrated in the drawing below. This system becomes an ultrafiltration system when the addition of distilled water is halted to allow concentration of the emulsion. Low molecular weight impurities may be removed from photographic emulsions employing spiral wound cellulose acetate or polysulfone membranes. Prior to or after diafiltration, ultrafiltration can be used to remove water from the emulsion in order to concentrate to the optimum diafiltration or coating concentration.

The following examples illustrate the practice of this disclosure utilizing an Osmonics SEPA type 0 spiral wound cellulose acetate permeator:

#### Example I

A 50 US gallon batch of a photographic silver halide emulsion was ultrafiltered and continuously diafiltered for 20 hours. The operating temperature and pressure were 38°C and 50 psi, respectively. Table I shows the metal ion and nitrate content of the permeate removed from the emulsion as the run progressed.

#### Example II

A 50 US gallon batch of a photographic silver halide emulsion was continuously diafiltered for 5.5 hours. The operating pressure and temperature were 50 psi and 43°C respectively. Table II shows the metal ion content of the permeate as the run progressed.

**TABLE I**

Effect of Continuous Diafiltration on Low Molecular Weight Solutes  
(50 US Gallon Batch)

Process	Operating Time (Hours)	Approx Gelatin Conc (%)	Flux (ml/min)	Metals-Permeate (ppm)			Nitrate (ppm)
				Ag	Na	K	
UF*	Start	0.9	1050	0.66	292.0	39,250	5100.0
UF	4.7 (end UF)	5.0	510	0.51	290.0	33,900	7000.0
DF	8.1	5.0	570	0.01	4.5	1,270	2100.0
DF	8.9	5.0	610	0.01	3.4	380	70.0
DF	10.4	5.0	600	0.01	2.4	---	26
DF	11.6	5.0	600	0.01	1.0	26	4.3
DF**	22.2	5.0	680	0.01	0.6	5	0.1

\*Ultrafiltration

\*\*Diafiltration

**TABLE II**

Diafiltration of Emulsion  
(50 US Gallon Batch)

Operating Time (Hours)	Flux (ml/min)	Metals-Permeate (ppm)		
		Ag	Na	K
Start	600	0.15	1400	5542
0.75	460	0.06	638	2386
1.00	480	0.04	315	1137
2.75	520	0.01	26	109
3.75	540	0.01	9	27
5.50	550	0.09	3	10

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