

Emulsion dopant recipe

Source: <https://patents.google.com/patent/EP0556715A2/en>

Solution A

gelatin	101.7g
n Sodium polyisopropylene-polyethyleneoxy-disuccinate (10% methanol solution)	30ml
Acetic acid(56%)	93.3ml
Ammonia (28%)	155.4ml
Silver bromide seed emulsion (average grain size 0.240 u.)	0.1465 mole equivalent
Pure water was added to 10593.0 ml.	

Solution B

Silver nitrate	1775.1g
Ammonia (28%)	1448 ml
Pure water was added to 4975.5 ml	

Solution C1

Gelatin	33.2g
Potassium bromide	406.8g
Potassium iodide	11.58g
Pure water was added to 1660.8 ml	

Solution C2

Gelatin	66.3g
Potassium bromide	811.9g
Potassium iodide	23.11g
Pure water was added to 3314.7 ml	

Solution D

Acetic acid(56%)	3000ml
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Solution E

Potassium bromide	208g
Pure water was added to 500 ml	

41% solution

Preparation of comparative emulsion Em-1

With the temperature of solution A kept at 40 °C, and while it was thoroughly stirred, solutions B, C1 , and C2 were added at rates shown in Table 1 using the double-jet method. During addition, solutions D and E were used to control pH and pAg as shown in Table 1 . The obtained emulsion was washed and dispersed using a usual method and the resulting emulsion was named Em-1. Electron microscopic observation revealed that the emulsion grains were regular octahedral grains having an average size of 1.00 urn.

Em-12 was prepared in the same way as Em-1, except that 93.7 mg of lead nitrate and 44.8 mg of potassium ferrocyanide were added to solution C1 , and 187.0 mg of lead nitrate were added to solution C2.

Time	Addition rate of solution B (ml/min.)	Addition rate of solution C ₁ (ml/min.)	Addition rate of solution C ₂ (ml/min.)	pH	pAg
0'00"	3.38	3.38		9.00	9.70
11'38"	5.43	5.43		9.00	9.70
19'48"	7.24	7.24		9.00	9.70
26'05"	9.10	9.10		9.00	9.70
31'10"	11.10	11.10		9.00	9.70
35'24"	12.98	12.98		9.00	9.70
42'09"	17.12	17.12		9.00	9.70
49'37"	23.52	23.52		9.00	9.70
60'20"	32.47	32.47		8.93	9.80
74'54"	42.25	42.25		8.81	10.00
81'52"	44.22	44.22		8.74	10.10
81'53"	44.22		44.22	8.74	10.10
85'10"	44.92		44.92	8.71	10.16
100'56"	43.16		43.16	8.56	10.40
111'04"	45.40		45.40	8.46	10.42
121'58"	46.08		46.08	8.34	10.44
155'50"	41.49		41.49	8.01	10.50
157'02"	41.49		41.49	8.00	10.50

Recipe scaled down and modified

Recipe has been adjusted to match scale of 20g of silver nitrate = 0.11773 moles

$$20/1775.1=0.011266 \text{ scale factor}$$

$$\text{Example: gelatin } 101.7 \times 0.011266 = 1.14\text{g}$$

Solution A

gelatin	1.14g
Acetic acid(56%)	1.05ml
Ammonia (28%)	1.75ml
Silver bromide seed emulsion (average grain size 0.240 u.)	$1.65 \times 10^{-3} \text{ mol}$
Pure water	119.3ml

Solution B

Silver nitrate	20g
Ammonia (28%)	16.3ml
Pure water	56ml

Solution C1

Gelatin	0.37g
Potassium bromide	4.58g
Potassium iodide	0.13g
lead nitrate	$1.055 \times 10^{-3} \text{g}$
potassium ferrocyanide	$5.047 \times 10^{-4} \text{g}$
Pure water was added to 1660.8 ml	18.7ml

Solution C2

Gelatin	0.75g
Potassium bromide	9.14g
Potassium iodide	0.26g
lead nitrate	$2.106 \times 10^{-3} \text{g}$
Pure water	37.3ml

Measuring out precise weights of modifiers

Dopants will be used in solutions of 0.1% or 0.05%

For solution C1:

Lead nitrate: $1.055 \times 10^{-3} \text{g} / 0.1\% = 1.05 \text{ml}$ of 0.1% solution

potassium ferrocyanide: $5.047 \times 10^{-4} \text{g} / 0.05\% = 1 \text{ ml}$ of 0.05% solution

For solution C2:

Lead nitrate: $2.106 \times 10^{-3} \text{g} / 0.1\% = 2.1 \text{ml}$ of 0.1% solution

0.1% solution is prepared by adding 0.1g of dopant to 100ml(g) of water or
1g/1000ml(g)

0.05% solution : 0.1g/200ml

Sensitizers

Hypo: 100mg/mole of silver $\rightarrow 0.1 \times 0.11773 = 0.01173 \text{g} / 0.58\% = 2 \text{ml}$

Steigmanns gold solution:

Stock: 0.5ml 1% gold chloride into 4.1ml 1% ammonium thiocyanate

dilution: 2ml stock into 15.5ml water, use **8.8ml**

Anti-foggants

KBr: 849mg/mole of silver $\rightarrow 0.849 \times 0.11773 = 0.09995 \text{g} / 10\% = \sim 1 \text{ml}$

Dyes

Erythrosine: 100mg/mole of silver $\rightarrow 0.1 \times 0.11773 = 0.01173 \text{g} / 1\% = 1.1 \text{ml}$

Tartrazine

Eosin

Hardener

Chrome alum: 3% to amount of gelatin, generally use **1ml** of 6%/25ml emulsion

Surfactant

Ethanol: 2.5ml/g of silver

SLES: unknown

Preservative

Thymol: 1 drop 10% solution/ 100ml emulsion

Coating density

4mg/cm² AgBr (20g silver nitrate gives 22.1g AgBr)

9-10ml/100cm²->0.1ml/cm²

Working out final volume of emulsion $22.1\text{g}/4\text{mg}=5525 \times 0.1 = \sim 552\text{ml}$ of emulsion

10% gelatin-> $552 \times 10\% = 55.25\text{g}$