Preparing Mixtures & Solutions: Definitions, General Concepts, and Calculations

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Definitions

The terminology used to describe adding a compound(s) to another compound(s) is confusing. Here are a few definitions (terms in **bold** are also defined):

- **coagulation**: the thickening of a **solution** due to a physical change (e.g., heating) or chemical change (e.g., addition of acids or alkalis).
- colloid: A substance present in solution in the colloidal state.
- **colloidal solution**: a **solution** in which the **solute** is present in the **colloidal state**. Examples include starch paste, gelatin glue, methylcellulose adhesive, PVAs, etc. The **solvent** is termed the **dispersion medium** and the dissolved substance the **disperse phase**. Several types of colloidal solution are possible, depending upon whether the dispersion medium and the disperse phase are respectively liquid and solid (suspensoid sols), liquid and liquid (emulsoid sols), gas and solid, etc. If the disperse phase, when removed from the solution by **evaporation** or **coagulation**, returns to the colloidal state on merely mixing with the dispersion medium, it is termed a reversible or lyophilic (solvent-loving) **colloid**, e.g., methylcellulose. If the disperse phase does not return to the colloidal state on simple mixing, it is termed an irreversible or lyophobic (solvent-heating) colloid, e.g., PVA.
- **colloidal state**: a system of particles in a **dispersion medium**, with properties distinct from those of a true **solution** because of the larger particle size.
- **compound**: a substance consisting of two or more elements chemically united in definite proportions by weight.
- disperse phase: The dissolved or suspended substance in a colloidal solution or suspension.
- **dispersion**: a **disperse phase** suspended in a **disperse medium**. A system of particles dispersed and suspended in a solid, liquid, or gas.
- **dispersion medium**: a medium in which a substance in the **colloidal state** is dispersed; the **solvent** in a **colloidal solution**.
- **emulsion**: a two-phase system in which the **disperse phase** consists of minute droplets of liquid, e.g., mayonnaise is an emulsion of oil and an aqueous compound, such as vinegar or lemon juice, made possible by the introduction of an emulsifier, such as lecithin found in egg yolks.
- evaporation: the conversion of a liquid into vapor without necessarily reaching the boiling point.

mixture, mechanical: these mixtures differ from molecular mixtures (solutions) and chemical **compounds** in the following respects:

- constituents may be separated by physical or mechanical means
- most mixtures may be made in all proportions; **solutions** often have limited solubility, e.g., **saturated solutions**
- no heat effect is produced on formation of mixtures
- properties of a mixture are an aggregation of the properties of all of the constituents

saturated solution: a **solution** that can exist in equilibrium with excess of **solute**, e.g., saturated calcium hydroxide solution.

solute: a substance that is dissolved in a solvent to form a solution.

- **solution**: a homogeneous molecular mixture of two or more substances of dissimilar molecular structure. Although the word is usually applied to solutions of solids in liquids, other solutions include: gases in liquids (solubility increases with a decrease in temperature); gases in solids; liquids in liquids; and solids in solids (e.g., some alloys).
- **solvent**: a substance (usually liquid) having the power of dissolving other substances in it. That component of a **solution** that has the same physical state as the solution itself, e.g., in a solution of sugar in water, water is the solvent while sugar is the **solute**.
- suspension: a two-phase system consisting of very small solid particles distributed in a liquid dispersion medium.

General Concepts

Percentage: The percentage of a solution or mixture refers to parts of one compound dissolved or mixed per 100 parts of the whole. To find the percentage or concentration of a compound in a solution or mixture, you need to know: (1) the weight or volume of the smaller component, and (2) the total weight or volume of the final solution or mixture. The percentage is calculated by dividing (1) by (2) and multiply the result by 100%.

Example: If 3 ounces of sugar are dissolved in enough water to make the total solution up to 8 ounces, the percentage of sugar dissolved in the total volume of the solution is determined in two steps: 1) $3 \div 8 = 0.375$

2) 0.375 x 100% = 37.5% sugar solution

Weight/Weight: Used primarily for two solids. Expressed as "w/w," this abbreviation usually describes a mixture made by the following actions:

- a. weigh out one solid (the smaller one),
- b. mix all of a. in a known weight of the other solid to bring the weight up to the desired total.

To determine the percentage of the solution or mixture, divide the weight of a. by b. and multiply the result by 100%.

Example 1: Mixing 10 g. (g. = grams) of solid A with 990 g. of solid B has a total weight of 1000 g. w/w. To find the solution percentage:

1) $10 \div 1000 = 10$

2) $10 \times 100\% = 10\%$

Example 2: If you dissolve or mix 10 g. of solid A in 100 g. of solid B, the total weight of the mixture is 110 g. Therefore, the percentage is 9.1%:

1) $10 \div 110 = 0.0909$ 2) $0.0909 \ge 100\% = 9.1\%$

Weight/Volume: Used primarily for a solid (solute) in a liquid (solvent). Expressed as "w/v" (or occasionally, "v/w"), this abbreviation describes a solution or dispersion made by the following actions:

- a. weigh out the solid,
- b. dissolve or mix the solid in a small volume of the liquid in a graduated container and add enough of the liquid to bring the total solution or dispersion up to the volume required.

To determine the percentage of the solution or mixture, divide a. by b. and multiply the result by 100%.

Example: Dissolve 10 g. of solid A in about 50 ml. (ml. = milliliters) of liquid B and bring the volume of the solution up to 100 ml. This is a 10% solution w/v: $(10 \div 100) \times 100\%$.

Volume/Volume: Used primarily for two liquids. Expressed as "v/v," this abbreviation describes a solution or an emulsion made by the following actions:

- a. measure out the volume of one liquid (solute) (usually the smaller one),
- b. add enough volume of the other liquid (solvent) to bring the total volume up to the amount required.

To determine the percentage of the solution or mixture, divide a. by b. and multiply the result by 100%.

Example 1: Adding enough of solvent B to 8 ml. of solute A to make a total solution volume of 850 ml. is a 0.94% solution v/v: $(8 \div 850) \times 100\% = 0.94\%$.

Example 2: An emulsion made of 6 ml. of oil, 95 ml. of vinegar, and 5 ml. egg yolk can also be described as a 5.7% oil in vinegar emulsion: ($6 \div 106$) x 100% = 5.66%

Ratios or Parts: Sometimes it is easier to describe solutions, emulsions, dispersions, or mixtures by using ratios or parts. This method is expressed by using a ratio, e.g., 1 part : 2 parts. Because there are often no specific weights or volumes indicated when this method is used to make a solution or mixture, it is important to denote whenever the measure was w/w, w/v, or v/v. To determine the percentage of the solution using a ratio:

1) add the numbers on either side of the : (colon) to arrive at the total number of parts,

2) divide the number of parts on the left of the : by the total number of parts,

3) multiply the result from 2) by 100%.

Example: A recipe for wheat-starch paste calls for 1 part of wheat starch to 4 parts of water, written 1: 4 v/v. This means that 1 part of the starch is measured out using a volume utensil, e.g., beaker, teacup, tablespoon, etc., up to some mark or known volume on the utensil. That same utensil is then used to measure out the water to the same mark or volume times the number of parts called for in the recipe, in this case, 4 parts of water. These two compounds are then mixed together, and although the starch does not dissolve in water, it does form a colloidal solution after cooking. The resulting concentration of the paste is 20% wheat starch in water:

1) total number of parts is 5

- 2) 1 part ÷ 5 parts = 0.20
- 3) 0.20 x 100% = 20%.

The paste can also be described as 20% solids. **NB**: During prolonged cooking, water is driven off as vapor, and the resulting paste has a higher % of solids, i.e., it is more concentrated, is a stronger adhesive, will shrink more, and be more brittle upon drying.

Molar solutions: Although not commonly used in the preparation of adhesives, molar solutions are sometimes referred to and knowing how to make them can be important. A molar solution is one that contains the molecular weight of a compound (in grams) in water (in liter(s)). To determine the molecular weight of a compound, you need to known the chemical formula of that compound as well as the atomic weight (or mass) of each of the elements in the formula (see the Appendix A). The atomic weight of each element is then multiplied by the number of molecules of that element in the formula. The sum of those numbers are the molecular weight (MW) of the compound. The compound is weighed out in grams to equal the MW. This is dissolved in about 500 ml. of water, and the solution is brought up to 1 liter with more solvent. This is a 1M (M =Molar) solution. (If a 5M solution is required, then 5 times the MW of the compound is weighed out in grams, dissolved in about 500 ml. of water, and the solution is brought up to 1 liter.)

Example: You need a 2M solution of sodium hydroxide, NaOH. The atomic numbers are:

- Na. sodium: 23
- H, hydrogen: 1
- O, oxygen: 16.
- Therefore, the molecular weight of NaOH is: 23 + 1 + 16 = 40. To make a 1M solution of NaOH, we would add 40 g. of NaOH to 500ml of water, and once dissolved, bring that solution up to 1 liter. To make a 2M solution, follow the same procedure, except dissolve $40g \ge 2 = 80 g$./liter.

Calculations for Diluting Stock Solutions

Calculation 1: To determine the concentration of a diluted solution when you know both the % concentration and starting volume of the stock solution:

1) $A = BC \div D$ 2) E = A - CSteps: A = total volume of the required solutionWhen: B = % concentration of the stock solution C = volume of the stock solution D = % concentration of the required solution E = unknown, amount of additional solvent required Example: To carry out a particular procedure, you require a 4% methylcellulose solution.

Unfortunately, there is only 200 ml. of a 5% stock solution made up. How much water needs to be added to the 200 ml. of the 5% methylcellulose solution to make a 4% solution? $\Lambda = unknown$ volume of required solution When[.]

when.	A – unknown, volume of required solution
	B = 5% concentration of the stock solution
	C = 200
	D = 4% concentration of required solution
of additiona	al solvent required Therefore : 1) $A = BC \div D$

E = unknown, amountA = 0.05 x 200 = 250

2)
$$E = A - C$$
 $E = 250 - 200 = 50$

Answer: 50 ml, of water has to be added to 200 ml, of the 5% stock solution to make 250 ml, of a 4% solution.

0.04

Calculation 2: There is another method for determining the % of a diluted solution using parts. $A = B \div (C + D)$ Step:

When: A = unknown, % concentration of the required solution

B = % concentration of the stock solution

- C = number of parts of the stock solution
- D = number of parts of the solvent used to dilute the stock solution

Example: A 30% stock solution is diluted down to a working consistency by adding 2 parts of solvent to 1 part of the stock solution. What is the % concentration of the diluted solution?

Known: A = unknown, % concentration of the required solution B = 30%

$$C = 1$$

D = 2

Therefore: $A = B \div (C + D)$ A = 30 = 101 + 2

Answer: The diluted solution is 10%.

Calculation 3: If math is not your forte, use the following table to:

• adjust the concentration of a stock solution to a lower concentration by diluting part(s) of the stock with part(s) of solvent, v/v.

• by keeping track of the number of parts of a solvent you add to known part(s) of a stock solution to arrive at the right working consistency, you can determine the % concentration of the solution.

% of soln.	stock →	40%	30%	20%	10%	8%	6%	5%	4%	3%	2%	1%
Parts of Stock	Parts of Solvent	¥	Ļ	Ļ	%	of	dilute	soln.	↓	↓	↓	Ļ
1	.25	32	24	16	8	6.4	4.8	4	3.2	2.4	1.6	.8
1	.50	26.6	20	13.3	6.7	5.3	4	3.3	2.6	2	1.3	.7
1	1	20	15	10	5	4	3	2.5	2	1.5	1	.5
1	1.5	16	12	8	4	3.2	2.4	2	1.6	1.2	.8	.4
1	2	13	10	6.6	3.3	2.7	2	1.7	1.3	1	.08	.3

1	3	10	7.5	5	2.5	2	1.5	1.25	1	.75	.5	.25
1	4	8	6	4	2	1.6	1.2	1	.8	.6	.4	.2
1	5	6.7	5	3.3	1.7	1.3	1	.83	.67	.5	.3	.17

Appendix A

Table of Symbols, Elements, Atomic Weights

Notes: Value given in parentheses denotes the mass number of the longest-lived isotope * Atomic weight of the most commonly available long-lived isotope

Ac	Actinium	227.03*	Co	Cobalt 58.	93		Ι	Iodine 126	90	
Ag	Silver 107.	.87	Cr	Chromium	51.	996	In	Indium	114	.82
Al	Aluminum	26.98	Cs	Cesium	132	2.90	Ir	Iridium	192	2.22
Am	Americium	(243.00)	Cu	Copper	63.	55	K	Potassium	39.	1
Ar	Argon 39.9	95	Dy	Dysprosiu	m	162.50	Kr	Krypton	83.	80
As	Arsenic	74.92	Er	Erbium	167	7.26	La	Lanthanum		138.90
At	Astatine	(210.00)	Es	Einsteiniu	n	(252.00)	Li	Lithium	6.94	4
Au	Gold 196.	.97	Eu	Europium	151	1.96	Lr	Lawrenciu	n	(260.00)
B	Boron 10.8	31	F	Fluorine	18.	998	Lu	Lutetium	174	.97
Ba	Barium	137.33	Fe	Iron 55.	85		Md	Mendelevit	ım	(258.00)
Be	Beryllium	9.01	Fm	Fermium		(257.00)	Mg	Magnesium	ı	24.30
Bi	Bismuth	208.98	Fr	Francium	(22	(3.00)	Mn	Manganese		54.94
Bk	Berkelium	(247.00)	Ga	Gallium	69.	72	Mo	Molybdenu	m	95.94
Br	Bromide	79.90	Gd	Gadoliniur	n	157.25	Ν	Nitrogen	14.0	01
С	Carbon	12.01	Ge	Germaniur	n	72.59	Na	Sodium	22.9	99
Ca	Calcium	40.08	Н	Hydrogen	1.0	1	Nb	Niobium	92.9	91
Cd	Cadmium	112.41	Ha	Hahnium	(26	52.00)	Nd	Neodymiur	n	144.24
Ce	Cerium	140.12	He	Helium	4.0	0	Ne	Neon 20.1	8	
Cf	Californiun	n (251.00)	Hf	Hafnium	178	3.49	Ni	Nickel 58.6	9	
Cl	Chlorine	35.45	Hg	Mercury	200).59	No	Nobelium	((259.00)
Cm	Curium	(247.00)	Ho	Holmium	164	4.93	Np	Neptunium	237	.05*

O Oxygen 15.999 **U** Uranium 238.03

190.2

V Vanadium 50.94

Tungsten 183.85

Ytterbium 173.04

Zirconium 91.22

Zinc 65.38

88.91

Xenon 131.29

Yttrium

W

Xe

Y

Yb

Zn

Zr

- **P** Phosphorus 30.97
- Pa Protactinium 231.04*
- **Pb** Lead 207.2

Osmium

Os

- Pd Palladium 106.42
- **Pm** Promethium (145.00)
- **Po** Polonium (209.00)
- Pr Praseodymium 140.91
- Pt Platinum 195.08
- Pu Plutonium (244.00)
- **Ra** Radium 226.02*
- Rb Rubidium 85.47
- Re Rhenium 186.21
- Rf Rutherfordium (261.00)
- Rh Rhodium 102.90
- **Rn** Radon (222.00)
- Ru Ruthenium101.97
- S Sulfur 32.06
- Sb Antimony 121.75
- Sc Scandium 44.96
- Se Selenium 78.96
- Si Silicon28.08
- Sm Samarium 150.36
- **Sn** Tin 118.69
- Sr Strontium 87.62
- Ta Tantalum 180.95
- **Tb** Terbium 158.92
- Tc Technetium (98.00)
- Te Tellurium 127.60
- **Th** Thorium 232.04*
- Ti Titanium 47.88
- TI Thallium 204.38
- Tm Thulium 168.93

Appendix B

Miscellaneous Conversion Factors

To convert	into	multiply by	Reciprocal
grains	grams	0.0648	15.432
ounces	grams	28.35	0.0353
pounds	grams	453.6	0.0022
inches	centimeters	2.54	0.3937
feet	meters	0.305	3.281
gallons	liters	4.54	0.22
gal	cubic feet	0.160	6.25
cubic feet	liters	28.38	0.3524
pounds/gallon	kilograms/liter	0.0998	10.022
sq. yards	sq. meters	0.8361	1.196
gallons	cubic meters	0.00455	220.0

Note: To reverse the conversion, multiply by the Reciprocal.

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Adhesives for Book Artists and Conservators Handout

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Guild of Book Workers • Standards of Excellence

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INTRODUCTION

While this publication is primarily intended for use by conservators, bookbinders, bookmakers, and book and fine artists, using paper-based materials, as well as leather and cloth, should find the information useful in understanding those adhesives they either currently use or want to use. While the overriding concern in presenting this information centers around two properties of materials – *stability* over time (both the adhesive and the materials it contacts) and *reversibility*, the author, who is a retired professional paper/book conservator with more than fifty years' experience, acknowledges that these may not be as important to creators of artifacts. Nevertheless, the use of good-quality, stable materials need not compromise the creative process. Therefore detailed information about a wide range of adhesives, e.g., both water- and organic-solvent-soluble, are presented to allow the reader to make informed choices no matter whether s/he is concretely expressing an idea or conserving a valued or valuable artifact.

The benefit of making up adhesives at home or in the conservation lab vs. purchasing ready-to-use adhesives is obvious. If you make the adhesive, you have complete control over the selection of ingredients, how it is prepared, and its working properties. Remember that practical experience using adhesives is key to successful and repeatable work. With the information presented herein and your ingenuity, you can perform almost any task with just a few good-quality adhesives that you can make yourself.

While many commercial adhesives are noted by their manufacturers or distributors as being "archival" and having passed the "photo activity test," the essential problem remains that *their components are propriety, i.e., secret and liable to change at any point without notification.* ("Archival" and other catch-phrases are discussed in the next section: What Does that "Term" Mean?) The "better" commercial products can probably be safely used on secondary materials, e.g., PVA, but their application to primary materials is generally not recommended.

When using an adhesive, e.g., starch paste, methylcellulose, sodium carboxymethylcellulose, or mixtures of these for a particular task, it is as important to understand their differing properties, including purity, viscosity, and concentration. For example, there are different viscosity grades and purities of methylcellulose on the market and results from the use of one may differ substantially from another, leading to confusion and frustration. These properties, along with choosing the best methods of application and optimum drying environments, will contribute to successful and repeatable results.

Purity: Purchase adhesive ingredients in the most pure form available (pharmaceutical grade is best, followed by food, technical, and industrial). Most of the adhesives listed below are repackaged and sold through suppliers, and information about purity, viscosity grade, etc., may not be readily available; try to get this information from the distributor. A good rule is not use low-purity-grade adhesives on primary paper and boards as they tend to deteriorate due to exposure to air, light, gaseous pollution, heat, and/or moisture.

Viscosity: The viscosity of an adhesive – the thickness of the adhesive in solution in a particular solvent at a standard concentration and temperature – is a property that few users apply informatively to their work. Generally, viscosity is linked to molecular weight (MW). The higher the MW, the longer the molecular chain; high MW materials are much less likely to degrade under normal conditions. MW is also linked to the degree of polymerization (DP). Changes in DP describe the shortening of the molecular-chain length after exposure to air, chemical(s), heat, light, and/or atmospheric pollutants. Materials with high MW and DP values form inherently viscous solutions and are generally more stable than materials with low MW and DP values. However, one should not choose a material with high intrinsic viscosity on the assumption that, if it is made up in a variety of thick to thin solutions, it will solve all problems. For example, an adhesive with long polymer chain length, e.g., METHOCEL A4M, cannot penetrate into a porous surface like paper no matter what the concentration is because the chains are so long, they get entangled and remain on the paper surface. Instead choosing METHOCEL A4C would penetrate into the paper better because the polymer chain length is 10 times shorter than A4M.

Concentration: Concentration is the amount of one or more compounds in the entire solution. If a solid is dispersed or suspended, but not dissolved, in a liquid, it is often labeled as % solids, e.g., high solids PVA.

The following table includes adhesives covered in this handout. Most of the recommended adhesives are aqueous solutions and are "reversible" in water. They are listed in order of stability, BUT it is really important to understand that these materials, especially the cellulose ethers, are NOT

interchangeable in terms of their properties and effectiveness in solving particular problems. It is essential that (1) the user determine what problem needs to be solved, (2) select the best type of material for the job, and (3) chose the most stable brand name among that type.

ations & es
: weak , use as) stronger s; 2–4%: leanser, lelivery onsolidant
ng agent, anser; do n adhesive as it is too ik!
nmended l grade)
lhesive, leanser, solvent- ubstrate, idant
nmended l grade)
ethanol: it for red- 6 below
iii iii iii iii iii iii iii iii iii ii

	rice starch • Remy DR Wheat Paste #301 is a pre-cooked, modified wheat starch paste in flake	rice starch • Beneo, Belgium • ADM		For conservation use, avoid pre-cooked starch paste powder.
	form that merely has to be dissolved in water before use.	PAYGEL 290		for conservation, though it might be useful as a binder for making paste papers.
Remoisten- able adhesive systems	Japanese paper coated with: • Aqualon CMC 7H3SF Pharma • "The Mix" (wheat starch paste and MC)	see recommended adhesives above	C/A	adhesives; concentration as for specific adhesive listed above, or based on use
Polyvinyl acetate (PVA) dispersions	 Jade 403 (403N) Jade 711 other good-quality PVAs (avoid ones with strong smell) 	Jade: Aabbitt Adhesives	A/book repair	adhesive: as purchased or dilute with water or METHOCEL A4M
Ethylene vinyl acetate (EVA)	• BEVA 371 Film •BEVA 371 Gel •BEVA 371 Solution	• BEVA: Conservator's Products Co.	C/A	These are organic- solvent-activated and heat-activated adhesives for linings, repairs, hinging, collage.
Protein glues	 gelatin, photographic or pharmaceutical grade gelatin, food grade parchment glue isinglass (from sturgeon), with no plasticizer added 	 Kodak Rousselot Co. Knox parchment scraps search Internet for supplier 	C/A A C/A C/A	 3%: sizing agent 1–3%: consolidant as above use based on tackiness use based on tackiness

Glue sticks	• Scotch Permanent Glue Stick • UHU Stic		Α	adhesive; avoid any glue stick that has a noticeable smell; test pH before using, should not be above pH 8.5
Acrylic co- polymers	• Rhoplex AC33 • Rhoplex N–580	Dow Chemical	• C/A • C (secondary materials)/A	 adhesive; heat-seal contact adhesive
Dry mount sheets and sprays	see p. 13			adhesive; do not use on or near original artifacts

CELLULOSE ETHERS

Description: Cellulose ethers are synthetic materials obtained by chemically modifying high-alpha cellulose with a variety of compounds. Such ethers have vastly different purity grades, viscosities, solvent solubilities, etc. These differing characteristics make them useful as adhesives, as well as binders, surfactants, coatings, sizing agents, consolidants, fixatives, gel media, solvent-delivery substrates, surface cleansers, etc. If made with deionized or distilled water and not contaminated, pharmaceutical-grade cellulose-ether solutions have long shelf lives and need no refrigeration.

Many of the characteristics of a cellulose ether, e.g., whether soluble in organic solvent or water, are dependent on the type of ether group and the degree of substitution (DS) of the group onto the –OH on the cellulose unit. Because it is difficult for the DS to be exactly planned or determined, manufacturers usually give a range of DS values for cellulose ethers. Since the DS values can vary considerably from batch to batch, the same cellulose ethers purchased in different batches may have slightly different properties, such as viscosity.



Methylcellulose, for example, is made by first treating native cellulose (high-alpha cellulose) with a strong alkali such as sodium hydroxide, NaOH. (\mathbf{R} = rest of cellulose ring)

Step 1. R–OH + NaOH = R–ONa + H₂O native sodium

alkali water

cellulose hydroxide cellulose

In Step 2, the alkali cellulose becomes methylcellulose when it is treated with methyl chloride, CH₃Cl.

Step 2.	R–ONa	+	$CH_3Cl =$	R-OCH ₃	+	NaCl
	alkali		methyl	methylcellulos	se	sodium
	cellulose		chloride			chloride

Generally, the higher the degree of polymerization (DP) number, the longer the polymer chains, and the greater the viscosity of cellulose ethers. Also, it is generally true that the higher the DP of a polymer, the more stable it is to aging. The viscosities of cellulose ethers are therefore controlled by altering the DP of the native, high-alpha cellulose *before* etherification. Experiments have indicated that some artificially aged cellulose ethers are very stable and compare favorably to similarly aged, such adhesives such as precipitated starch paste; only the flexibility of their dried films substantially differs.¹

Although many assume that cellulose ethers are similar, in fact they are quite different, except their pH. For example, methylcellulose (MC) and sodium carboxymethylcellulose (SCMC):

Methylcellulose	Sodium Carboxymethylcellulose				
non-ionic	ionic				
poor bonding strength	good bonding strength				
powder/fil insoluble in wrter ≫80°C	powder/fil soluble in cold/hot water				
high-surface activity: "wet"	low-surface activity: "dry"				
neutral pH	neutral pH				

Dow Chemical – the manufacturer of METHOCEL A, the most highly recommended methylcellulose – states that the viscosity of METHOCEL A4M, measured in a 2% solution of water at 20°C, is between 3000–5000 cPs (centipoise), thus "4M" is designated as having an average viscosity of 4000 cPs. Because viscosity is linked to degree of polymerization (DP), METHOCEL A4M forms long chains of methylcellulose in solution that become increasingly entangled as the concentration increases (2.5–4%). When applied to paper as an adhesive (or a binder for paste papers), the polymer is so tangled that it cannot penetrate far into the paper. However, because of MC's high-surface activity, the water is readily absorbed into the paper is dry, much of the polymer film forms on the surface to which it was applied. This film often produces a glossy or sparkling effect on the paper's surface.

Since METHOCEL A4M has poor adhesive power but excellent stability and reversibility characteristics, mixing it with stronger adhesives – starch paste or PVA – helps those dried films shrink less, and increases flexibility and reversibility. An all-purpose adhesive, known as "The Mix," can be made by mixing 1 volume of 17–20% undiluted wheat starch paste in 2 volumes of 2.5% METHOCEL A4M. Another practical advantage of this mixture is that "The Mix" remains fresh if stored in a closed container in the refrigerator for long periods. To increase working time and "slippage" with PVA, mix equal volumes of 2.5% METHOCEL A4M and PVA.

Many people making paste papers like to use methylcellulose as the binder because of its workability and because its solution does not spoil. However, as mentioned above, methylcellulose has poor bonding power. Sodium carboxymethylcellulose would be a far better binder to use for paste papers. However, because both MC and SCMC are hygroscopic, the dry paste may block – stick to – with another material if the RH is very high. For these reasons, "The Mix" or a good-quality cake flour might be a better choice for paste papers.

Purchasing a cellulose ether without knowing the following information might lead to disappointing

¹. Baker, Cathleen A. "Methylcellulose and Sodium Carboxymethylcellulose: An Evaluation for Use in Paper Conservation through Accelerated Aging." In *Preprints of the Contributions to the Paris Congress, 2–8 September 1984: Adhesives and Consolidants.* Eds. Norman S. Brommelle, Elizabeth M. Pye, Perry Smith, and Garry Thomson, 55–59. London: International Institute for Conservation, 1984; Feller and Wilt, *Evaluation of Cellulose Ethers.*

and inexplicable results. Suppliers should publish, or at least make available upon request, information about the exact type of cellulose ether, purity, viscosity (cPs), brand name, and date of manufacture. With regard to purity, pharmaceutical grade is strongly recommended, food grade is acceptable, but neither industrial nor technical grade is recommended. If you can determine the manufacturer, go to the Internet or write/call for technical information, the SDS for that product, instructions for making solutions, and by all means, request samples of the various types and viscosities available. Experiment with these samples by varying the concentrations to see how different and useful these materials can be.

Note: The popular leather consolidant Klucel G (hydroxypropylcellulose) is soluble in water or alcohol. However, its use should be restricted to situations when a more stable type of cellulose ether or other adhesive cannot be used. Feller and Wilt found that Klucel G is not as stable as either high purity methylcellulose or sodium carboxymethylcellulose.

One last note: While one person might praise a cellulose ether for a certain application, another may condemn it. Chances are they are not using the same purity grade and/or viscosity grade of a particular cellulose ether, or perhaps, two entirely different cellulose ethers, e.g., MC and SCMC.

Recipe: 2.5% methylcellulose: METHOCEL A4M

25 g. METHOCEL Å4M

975 ml. deionized or distilled water Hint: Pre-chill two-thirds of the water.

- 1. Weigh out the powder, wearing a dust mask, if necessary.
- 2. Heat a third of the water to boiling. Take it off the heat and allow to cool slightly.
- 3. Pour the hot water into the clean, air-tight storage container. While whisking, slowly pour in the powder. The powder will not dissolve but will disperse in the hot water. Whisk until all of the powder is dispersed.
- 4. Immediately pour in the remaining chilled water, whisking gently in order to evenly wet the powder. As soon as the solution starts to thicken, stop whisking – to continue will cause foaming. Stir occasionally over the next hour. To hasten the dissolution of the powder, cover and place the container in the refrigerator. After an hour or so, the solution will be ready for use without lumps.
- 5. If made with pure water and not subsequently contaminated, METHOCEL A4 solutions will keep without refrigeration.

Recipe: 2–3% methylcellulose: METHOCEL A4C

20–30 g. METHOCEL A4C

980–970 ml. deionized or distilled water Hint: Pre-chill two-thirds of the water.

- 1. Weigh out the powder, wearing a dust mask, if necessary.
- 2. Heat a third of the water to boiling. Take it off the heat and allow to cool slightly.
- 3. Pour the hot water into the clean, air-tight storage container. While whisking, slowly pour in the powder. The powder will not dissolve but will disperse in the hot water. Whisk until all of the powder is dispersed.
- 4. Immediately pour in the remaining chilled water, whisking gently in order to evenly wet the powder. As soon as the solution starts to thicken, stop whisking – to continue will cause foaming. Stir occasionally over the next hour. To hasten the dissolution of the powder, cover and place the container in the refrigerator. After an hour or so, the solution will be ready for use without lumps.
- 5. If made with pure water and not subsequently contaminated, METHOCEL A4 solutions will keep without refrigeration.

Recipe: 2.5 or 4% sodium carboxymethylcellulose

25 or 40 g. Aqualon CMC 7H3SF Pharma 975 or 960 ml. deionized or distilled water, room temperature

- 1. Fill a clean, air-tight storage container with all of the water.
- 2. Weigh out the powder, wearing a dust mask, if necessary.
- 3. While slowly pouring the powder into the water, whisk constantly. When all of the particles are in suspension, set aside and stir occasionally. The solution should be ready in about an hour or two with no lumps.

4. If made with pure water and not subsequently contaminated, these solutions will keep without refrigeration.

STARCH PASTES

Description: Starch and gluten are the two primary components of flour processed from most plant seeds, roots, and tubers. Precipitated pure starch is prepared from flour in a process that separates the carbohydrate starch from the proteinaceous gluten. Compared to flour pastes, such as from wheat, which in old paste recipes could also have contained alum and powdered rosin, paste made from precipitated starch is more stable and is less likely to discolor on aging. Starch contains both amylose (a straight chain polymer of alpha-linked glucose units) and amylopectin (a branched chain of repeating glucose units with branches every 25 units). Wheat and rice starch tend to have more amylose, which is more stable than amylopectin, than other starches.

Starch is insoluble in water and must be heated to burst the grains, absorb water, and thicken before it is used as a paste. Generally, the longer paste is cooked, the greater its adhesive/bonding power, primarily because the water evaporates and the paste becomes more concentrated. Therefore, the strength of a starch paste is based on its cooking time, as well as on its pre-cook concentration. *Unless* made in low concentrations and cooked minimally, paste should be diluted considerably before it can be used over large areas. Undiluted in high concentrations and cooked for more than 5 minutes after boiling, wheat-starch paste's adhesive properties, brittleness, and degree of shrinkage are great. (Such dried films are known to have pulled the Teflon coating off pans and delaminate the bottom of glass containers.) Low-concentration (less than 20%), minimally cooked (no more than 5 minutes after boiling), "dry" paste can be used undiluted as a mending or "collage" adhesive *if* brushed out as a very thin layer.

Note: The so-called "old" and "new" pastes are used in traditional Japanese mounting procedures. For the adhesion of new silk to paper supports, mounters use freshly prepared "new" paste, similar to the recipe given below. New paste is diluted down considerably with water before use because it is very strong. "Old" paste is used exclusively on the original paper support and its paper linings. This paste is aged 10–20 years before use, and during that time, almost all of its adhesive properties are lost through bacterial action. In fact, it acts more as a sizing agent for the linings than as an adhesive. The "adhesion" between the original paper and linings is due more to the raising of surface fibers with a special "pounding" brush to encourage the subsequent intertwining of fibers in the presence of "old" paste.

All starch pastes are naturally acidic. Conditioning the distilled water to pH 7 with calcium hydroxide $Ca(OH)_2$ before cooking will help buffer the acid in the paste, but it is important not to make it unnaturally alkaline.

Paste papers: Cake flour used in paste-paper decoration consists of both starch and gluten; the latter may cause some yellowing and possible degradation. However, centuries-old paste papers examined on a number of books, where less than pure flours were undoubtedly used, show little indication of failure – loss of adhesion to the paper. Therefore a good-quality cake flour used for this decorative technique can probably be considered a stable material.

Recommended starches:

- *zin shofu* (also *jin, zen, gin shofu*): a Japanese precipitated wheat starch
- AYTEX-P: a precipitated wheat starch (ADM)
- a precipitated rice starch (Beneo, Belgian)

Recipe: precipitated wheat or rice starch paste

- 1. First, condition deionized or distilled water to pH 7 with Ca(OH)₂.
- 2. Select a storage container just large enough to accommodate all of the paste and sterilize it by washing with soap and water, and rinsing it with a little ethanol or rubbing alcohol or hand sanitizer (the cover/lid, too). Wipe dry with paper toweling. (Using a larger container only traps more air inside and speeds up spoilage.)
- 3. Soak 1 volume of starch in 5 volumes of the conditioned water (17% soln.) or 4 vols. conditioned water (20% soln.) in a heavy pan, preferably non-stick, for several hours or overnight.
- 4. After soaking, cook on medium heat. Stir constantly throughout the entire cook.

- 5. Once it boils, the paste thickens and becomes translucent. After 4 minutes, it will begin to loosen. Cook for about 5 minutes from the boiling point. This is a relatively weak paste, which if used "thickly," is not as strong nor will it shrink as much as longer-cooked paste.
- 6. Immediately pour the hot paste into the sterile container. Pour just enough distilled or deionized water on top of the hot paste to completely cover it; this prevents a skin from forming on the surface as it cools to a gel. (If making rice-starch paste, a gel doesn't form after cooling.) *Do not refrigerate cooked paste because it negatively alters its adhesive and working properties.*
- 7. When the paste is cold, pour off the covering water. Using a hand-held blender wand or a food processor, blend the paste until smooth. If necessary, add a little conditioned water to make a smooth paste. You may also sieve the paste 3–4 times until smooth (don't use a metal sieve) and knead with a brush; add water to dilute to appropriate consistency.
- 8. Place plastic food wrap directly on top of the paste, cover, and store in a cool, dry place.
- 9. As paste is removed with a clean utensil (a popsicle stick is handy because you can throw it away after this one use), either move remaining paste into a smaller, clean container to slow down spoilage, or completely cover the surface of the paste by pressing a new piece of plastic wrap onto it. NEVER return any paste to the container.
- 10. Another storage option is to form the hot paste into a ball and place into a glass bowl. Cover the paste completely with deionized or distilled water to cool; no need to cover the bowl. Every day, throw away the old water and replace with fresh. This paste will keep for about a week without refrigeration.

"The Mix"

- 1. Put one volume of about 17–20% undiluted, freshly cooked, precipitated wheat-starch paste (blend, strain, or knead beforehand so that there are no lumps) into a clean container.
- 2. Using a "folding" action, blend two volumes of 2.5% METHOCEL A4M into the paste. Do not whip the mixture or use a blender as this will make the solution foam considerably.
- 3. Store in the refrigerator. If kept air-tight and free of contaminants, this mixture has been known to keep for a year. With the addition of the methylcellulose, the refrigerated starch-paste component of the "Mix" does not seem lose its working properties and bonding strength.

REMOISTENABLE-ADHESIVE REPAIR TAPES, HINGES, AND LININGS

Description: The very best repair tapes, hinges, and linings to be used on valued artifacts are homemade from good-quality Japanese paper and the appropriate adhesive depending on the situation, e.g., sodium carboxymethylcellulose, starch paste, "The Mix," or BEVA 371 Film or BEVA Gel.

Most of the commercially made, so-called "archival" tapes are based on pressure-sensitive adhesives of often undisclosed composition. Generally, such tapes are not recommended for application directly on any valued/valuable artifact; their use on secondary materials and for the repair of circulating library materials is probably fine.

Water-activated proprietary tapes, such as white "Holland," "Linen," or "Cambric" cloth, gummed tapes might be used to hinge heavy works of art, but are too bulky for repairing artifacts, even ones on heavy paper. The adhesive used for these tapes is probably dextrin, a modified starch paste. **Hint**: Saliva, rather than water, is best for activating this type of adhesive.

POLYVINYL ACETATE (PVA)

Description: Generally, the films of the polyvinyl acetate resins (in organic solvents) are more stable and reversible than the films of the polyvinyl acetate dispersions (in water), such as Jade 403. The reversibility of the dispersion films is very difficult, and at best, only swelling can be achieved with water, acetone, or mixtures of water and acetone. For this reason, PVA should not applied directly to the back of a book (spine) in a rebacking procedure. Instead apply paste or sodium carboxymethylcellulose to the back of the book, followed by a layer of Japanese paper adhered with one of those adhesives; when dry, apply PVA over this, if necessary.

A thicker PVA (Jade Thick) sets very quickly and is ideal for box-making. The amount of organic solvent in this dispersion may cause irritation for some users and should also be tested on the material(s) to be applied to for any change, such as color shift, bleeding, etc. **Warning**: Some years ago, the

preservative formaldehyde was removed from PVA solutions, and as a result they spoiled quite quickly, resulting in streaks of moldy discoloration. Therefore, purchase PVA in small quantities, but remember that if frozen, they will be irrevocably damaged and so should not be shipped in the winter months from/ to northern locations in the U.S.A.

Recommended PVA dispersions:

• Jade 403 (403N), Jade Thick; conservation-quality PVAs, also called "white glues"

ETHYLENE VINYL ACETATE (EVA) CO-POLYMERS

Description: Although not used extensively in the conservation of paper and books, these copolymers, when dispersed in an organic solvent or used as a film, can be indispensable, especially when water-based adhesives will not adhere to slick or greasy materials, such as Mylar, glass, or parchment. They are also useful when aqueous adhesives cannot be used, or when speed of application is required. They are usually used as heat-seal adhesives that require relatively low temperatures to activate, ca. 150°F. In gel or solution form, they are quick-setting and require little or no curing or fusing. Heat-seal tissues using EVA (especially BEVA products) can be easily made, stored for long periods, and appear to have good to excellent aging characteristics. Films of the solvent-soluble BEVA 371 Solution should be reversible in petroleum spirits; BEVA 371 Film in xylene; each is reversible with heat. The dried films of aqueous BEVA D–8 are reversible with heat and alcohol, while BEVA Gel films are reversible in water, toluene, xylene, propanol (rubbing alcohol), and ethanol. BEVA Gel can be used as a contact, as well as a heat-seal, adhesive.

So-called "reversible PVA" and "water-reversible PVA" are probably aqueously dispersed EVAs. They are not reversible in water to the point that they dissolve, and so, from a conservation viewpoint, probably should not be considered reversible in the same way that the recommended cellulose ethers are.

Recommended EVA, solvent-soluble:

- BEVA 371 Solution
- BEVA 371 Film (a cast, 1-mil film with no carrier; can be used as a double-sided "tape")

Recommended EVA, water-soluble:

- BEVA Gel
- BEVA D-8

PROTEIN GLUES

Description: Protein glues are derived by hydrolyzing in hot water fibrous protein collagen from any number of mammal or fish sources: skins, bones, hooves, horns, swim bladders, etc. Gelatin is a pure form of an animal glue. Gelatin is a natural polymer built from a variety of amino acids in a polypeptide-chain structure. Parchment glue is made by hydrolyzing parchment/vellum clippings. To maintain flexibility, proprietary liquid protein glues contain plasticizers and humectants such as sorbitol or glycerol.

Sheet/leaf or powder glues/gelatin dissolve in hot water, and the solutions set to a gel when cool. When using gelatin, it is important not to allow the solution to exceed 130°F (55°C). The color of less pure glue solutions, e.g., rabbit-skin glue or hide glue, is usually medium to dark brown. Gelatins are usually pale brown or off-white, while parchment glues are off-white in color. Like starch pastes, protein glues are naturally acidic, and to a certain extent, they act as pH buffers. Also as recommended for starch pastes, it is important not to allow protein adhesives to become too alkaline.

Recommended gelatins:

- gelatin, photographic and pharmaceutical grade: available through most chemical and photographic supply houses, e.g., Kodak, Rousselot
- parchment glue
- isinglass (from sturgeon), without plasticizer
- gelatin, food grade: Knox brand

Recipe: gelatin

- 1. For a 3% solution, weigh out 30 g. of gelatin. Add to 970 ml. of room-temperature deionized or distilled water.
- 2. Soak the gelatin in the water until the granules are swollen and soft.
- 3. Warm in a double-boiler until the solution reaches a temperature of 130°F (55°C), by which point all of the granules should be melted and dissolved.
- 4. Pour into a sterilized container and refrigerate. The solution will probably spoil in less than a week.
- 5. Dilute as needed by adding a little warmed water to the gelled solution and reheating it enough to make a workable solution; do not exceed 130°F (55°C).

Recipe: parchment glue

- 1. Cut scraps of parchment or vellum into small pieces, approx. 5–10 mm. square.
- 2. Soak the pieces in water (21 g. parchment/vellum to 2.5 liters water) and cook in a double-boiler at low heat for about 45 minutes. Do not let the mixture boil as impurities may be drawn from the skin. Strain through cheesecloth twice.
- 3. Cool the solution; the resulting stock will be a thick gel. Glue will keep for a few days in the refrigerator. Traditionally, vinegar was added as a preservative, but this is not recommended. It can be frozen for long-term storage.
- 4. To use: stir enough of the stock solution into warm water (35–50°C) to make a solution that is slightly tacky. To test for degree of tackiness: dip thumb and forefinger into solution, press together and let cool; the film should offer some resistance to the fingers as they are separated.

GLUE STICKS

Description: Although information about the chemical ingredients in glue sticks is scant, they seem to consist of polyvinyl pyrrolidone with polyalkylene polyamine. To the author's knowledge, no long-term study has been performed on glue sticks for use in paper or book conservation, but a "quick-and-dirty" aging test done over thirty years ago indicates that they age well and remain reversible in water for at least that time. While not recommended for conservation purposes, they are useful adhesives for tipping-in samples, hinging lightweight artwork, attaching labels, etc.

Unlike almost all other adhesives, glue sticks tend to be slightly to moderately alkaline. When they were first introduced to the market, they were actually too alkaline to be recommended for use, but since then, the formula has been changed to render them only slightly basic. If a brand smells strongly of ammonia, it should not be used. Recently UHU Stic Glue Stick was tested for pH: the glue was pH 7, and the glue dispersed in deionized water registered between pH 8.5 and 9.0.

Before use, it is strongly recommended that the glue stick be tested by applying and allowing it to dry thoroughly on a sample of any *colored or dyed* cloth, paper, or board to be used to insure that the alkalinity of the glue stick does not affect the color. Colored glue sticks that change to clear upon drying are not recommended because it is not known what the color- changing ingredient is.

Recommended glue stick:

- Scotch Permanent Glue Stick
- UHU Stic

ACRYLIC CO-POLYMERS

Description: These constitute a diverse range of acrylic resins with wet, dry heat-seal, or dry contact adhesive properties. Commercially, acrylic-resin dispersions form the binder in latex paints. Although the solutions are water-soluble initially, upon drying, their films are reversible in alcohols if fresh, and if aged films, in acetone. Heat-seal tissues have been made from both the resins and the dispersions, the latter being more common. The dispersion-based, heat-seal adhesives require sealing temperatures around 180–220°F making them less ideal compared to BEVA products, which seal at ca. 150°F. There also seems to be a potential problem with common acrylic-resin aqueous dispersions in that they are usually alkaline and not as stable as the resin dissolved in an organic solvent.

Note: In the past, the Library of Congress's so-called "LC Heat-Set Tissue" was made with a mixture of two different Rhoplex AC dispersions (ethyl methacrylate, methylacrylate co-polymer). (The term

"heat-set" is used incorrectly here as the adhesive is used as a dried film on tissue. It is activated and *sealed* with heat or solvent vapors; heat set refers to adhesives that need/produce heat to set initially, e.g., certain epoxies.) Crompton Coated Tissue is similar to the LC tissue; it is reversible with mineral spirits.

Rhoplex N–580 is a high MW, water-soluble acrylic dispersion (ethyl methacrylate/ methylacrylate co-polymer) that is very useful as a contact adhesive for secondary materials only, such as laminating boards together. Apply to the surface of one of two boards for a temporary bond, or apply to one surface of both boards for a permanent bond.

Recommended acrylic resin dispersions:

- Rhoplex AC33
- Rhoplex N–580 (do not use on original artifacts)

DRY MOUNT (HEAT-SEAL) SHEETS AND MOUNTING SPRAYS

Description: Like commercial tapes noted above, these materials are only recommended for use on secondary materials. Common brand names are: 3M Spray-Mount Artist's Adhesive 6065 Clear (preferred over 3M 77 Spray Adhesive); BufferMount Dry Mounting Tissue; Fusion 4000; Gudy 870 (Gudy O) and Gudy 831 (Gudy V); Rollataq Adhesive System; 3M Positionable Mounting Adhesive 568; Filmoplast R.

WHAT DOES THAT "TERM" MEAN?

Manufacturers and distributors will often use terms/phrases such as "archival quality," "photo activity test," "acid-free," and "buffered." These can be confusing, and most of the following definitions are paraphrased from the Society of American Archivists (SAA): <www.archivists.org/glossary/>; accessed October 2022.

Archival Quality: When pertaining to records-storage materials, archival-quality materials those that do not cause harm or reduce the life expectancy of the original, including adhesives. Testing organizations deprecate the use of "archival" because it is a highly subjective term, and the ubiquitous use of "archival" in commercial advertising to describe products suitable for materials and implying an infinite life span has made this use of the word nearly meaningless. When archivists need to specify media appropriate for records of enduring value or for storage containers for such materials, they avoid the term "archival" and instead use specific requirements such as lignin-free or acid-free with a 3 percent calcium-carbonate alkaline reserve.

Photographic Activity Test: (PAT, Photo Activity Test) International Organization for Standardization (ISO) 18916:2007, a test that checks for potential *chemical* reactions between photographs and the materials used to make enclosures for them. Developed by the Image Permanence Institute (IPI) at RIT, Rochester, N.Y., PAT is used to test papers, boards, adhesives, inks, paints, labels, tapes, etc. There are other tests that screen for potential *physical* damage such as blocking and abrasion.

Acid-free: Any material with pH 7 or greater *when manufactured*. While "acidic" has been a buzz word related to the stability of paper-based materials, its used has become, like "archival," fairly meaningless. What is often not considered is that many natural adhesives, such as starch paste and protein glues, are more stable in a slightly acid environment, ca. pH 6. Making them unnaturally alkaline makes them more unstable. Conservation-quality synthetic adhesives, such as the recommended water-soluble cellulose ethers, tend to be "naturally" pH neutral. To describe a non-aqueous adhesive as "acid-free" is meaningless as only water-based solutions can be measured for the concentration of either the hydrogen (H⁺) ion (pH 0–7, acidic to neutral) or hydroxyl (OH⁻) ion (pH 7–14, neutral to alkaline/basic). And be aware that a strongly basic material can be just as destructive to paper as a strongly acidic material.

Buffer: The SAA defines a buffer as a substance that can neutralize acids. Actually, true buffers neutralize both acids and bases. Calcium carbonate or magnesium carbonate – neither is a true buffer – is often added to paper (and some adhesives) to neutralize residual manufacturing acids or to protect materials from future acidic contaminants. **Note**: If you want to add either of these "buffers" in powder form to an aqueous adhesive, it will not dissolve unless the adhesive solution is below pH 6. Therefore, it is more efficient to add buffer liquids – saturated calcium hydroxide or magnesium bicarbonate – to adhesive solutions or to the water used to make up the adhesive. When dry, these buffers form carbonates, oxides, and hydroxides – alkaline reserves. Again, adding a buffer to a non-aqueous adhesive is not necessary because it does not have a "pH."

ASTM D–4236: For example, some glue sticks, e.g., Scotch Permanent Glue Stick (white cap) and UHU Stic, include on the packaging that they conform to ASTM D–4236 (American Society of Testing Materials), which means that they have been found to be non-toxic and suitable for use by children.

For more information, see accompanying handout: "Preparing Mixtures & Solution: Definitions, General Concepts, and Calculations."

GOOD LUCK!

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