

**Table II. Desiccant Efficiency in the Drying<sup>a</sup> of Various Amines<sup>b</sup>**

desiccant	residual water content, ppm		
	Et <sub>3</sub> N <sup>d</sup>	(Me <sub>2</sub> CH) <sub>2</sub> NH <sup>e</sup>	NH <sub>2</sub> (CH <sub>2</sub> ) <sub>2</sub> NH <sub>2</sub> <sup>e</sup>
KOH powder	37 (28) <sup>f</sup>	750 <sup>g</sup>	1370 (3700) <sup>g</sup>
4A sieves	33 (28) <sup>h</sup>	<25	<25
3A sieves	34	<25	<25
CaH <sub>2</sub>	68 (34) <sup>f</sup>	150 <sup>i</sup>	500 <sup>f</sup>
Na	83	<25	150
BaO	89 (53) <sup>f</sup>	50	1100
CaC <sub>2</sub>	98 (80) <sup>f</sup>	<25 <sup>i</sup>	<25 <sup>i</sup>
CaO	165 (56) <sup>f</sup>		
Al <sub>2</sub> O <sub>3</sub>	223 (223) <sup>f</sup>		
silica gel	451		
CaSO <sub>4</sub>		>2500	

**Table II. Efficiency of Desiccants in the Drying<sup>a</sup> of DMF<sup>b</sup>**

desiccant	residual solvent water content, ppm			
	6 h	24 h	72 h	other conditions
3A molecular sieves	500	187	98	1.5 <sup>c</sup>
P <sub>2</sub> O <sub>5</sub>	879	105	579	2 <sup>d</sup>
CaH <sub>2</sub>	641	227	102	94 <sup>d</sup>
4A molecular sieves	454	134	108	
KOH (powdered)	1360	1110		303 <sup>d</sup> 890 <sup>e</sup>
B <sub>2</sub> O <sub>3</sub>				
BaO	2060	1520	1140	
CaO	2090			
Al <sub>2</sub> O <sub>3</sub>	1970			
CaSO <sub>4</sub>	2310	2030	1420	
K <sub>2</sub> CO <sub>3</sub>	2500			

<sup>a</sup> Static drying modes unless otherwise specified. <sup>b</sup> Desiccant loading 5% w/v; initial water content 2860 ppm (0.286% w/w). <sup>c</sup> Sequentially dried sample, 72 h. <sup>d</sup> Distilled Sample. <sup>e</sup> Stirring for 24 h followed by distillation.

**Table IV. Efficiency of Desiccants in the Drying<sup>a</sup> of Acetone<sup>b</sup>**

desiccant	residual solvent water content, ppm			
	6 h	24 h	72 h	other conditions
B <sub>2</sub> O <sub>3</sub>				18 <sup>c,d</sup> 47 <sup>c,e</sup> 107 <sup>f</sup> 322 <sup>g</sup> 1700 <sup>h</sup>
3A molecular sieves	115	152	322 <sup>g</sup>	322 <sup>h</sup>
CuSO <sub>4</sub> (anhydrous)	1920	972	579	
4A molecular sieves	331	887	1720	
CaSO <sub>4</sub>	1590	1600		
BaO	1910	1870 <sup>i</sup>		
P <sub>2</sub> O <sub>5</sub>				1970 <sup>j</sup>
K <sub>2</sub> CO <sub>3</sub>	2057	2250		

<sup>a</sup> Static drying modes unless specified otherwise. <sup>b</sup> Desiccant loading 5% w/v; initial water content 2710 ppm (0.271% w/w), unless specified otherwise. <sup>c</sup> Initial water content 2890 ppm (0.289% w/w). <sup>d</sup> Stirred, distilled, and sequentially dried, 24 h. <sup>e</sup> Stirred for 24 h and distilled. <sup>f</sup> Dried for 24 h and then distilled. <sup>g</sup> Contamination (2%) by mesityl oxide. <sup>h</sup> Fractionated sample. <sup>i</sup> Contamination (12%) by mesityl oxide. <sup>j</sup> Brown-black solution.

**Table V. Comparison of Desiccant Drying Efficiency for Dioxane and Acetonitrile<sup>a</sup>**

desiccant	residual solvent water content, ppm	
	dioxane	acetonitrile
CaSO <sub>4</sub> <sup>b</sup>	240	180
CaCl <sub>2</sub> <sup>b</sup>	290	d
3A molecular sieve <sup>c</sup>	19	52
4A molecular sieve <sup>c</sup>	30	450

<sup>a</sup> Initial water content = 2500 ppm; drying time 72 h. Activation temperature: <sup>b</sup> = 225 °C. <sup>c</sup> = 350 °C. Drying temperature 27-30 °C. <sup>d</sup> CaCl<sub>2</sub> induces a base-catalyzed tritium exchange with acetonitrile which precludes determination; <sup>e</sup> desiccant loading = 5% w/v.

**Table II. Desiccant Efficiency in Drying<sup>a,b</sup> of 1,2-Ethanedithiol<sup>c</sup>**

desiccant	residual water content, ppm	
	6 h	72 h
3A sieves (bead)	1900 (1200, <sup>d</sup> 540 <sup>e</sup> )	
3A sieves (powder)	360 <sup>f</sup>	
4A sieves (powder)	1900 (2070) <sup>d</sup>	
MgSO <sub>4</sub>	3600	
CaC <sub>2</sub>	990 <sup>g</sup>	
B <sub>2</sub> O <sub>3</sub>	k	
BaO	k	
CaO	k	
distillation <sup>h</sup>	1080	65 <sup>h,i</sup> (76) <sup>h,j</sup>
benzene azeotrope	150 (76) <sup>h,k</sup>	
Mg	400	
Al		

<sup>a</sup> Static drying modes unless specified otherwise. <sup>b</sup> Water content assayed by the Karl Fischer method. <sup>c</sup> Initial water content 1500 ppm. <sup>d</sup> Initial water content 1000 ppm. <sup>e</sup> Initial water content 1030 ppm. <sup>f</sup> 96-h drying period. <sup>g</sup> 168-h drying period. <sup>h</sup> Analysis was performed after settling of desiccant, 3-6 h. <sup>i</sup> Weight of magnesium in accord with general practice, i.e., 0.5% w/v. <sup>j</sup> See text. <sup>k</sup> Weight of magnesium 2% w/v. <sup>l</sup> Distilled sample. <sup>m</sup> Initial water content 1670 ppm, distilled sample. <sup>n</sup> Weight of sodium 3% w/v. See ref 32. <sup>o</sup> Ratio of sodium to 2-butyl succinate for 2-BuOH and to diethyl phthalate for ethanol in accord with general practice (see ref 7c), i.e., Na, 0.3 mol L<sup>-1</sup>; dicarboxylic acid ester, 0.14 mol L<sup>-1</sup>. <sup>p</sup> Stirred sample. <sup>q</sup> No apparent drying.

**Table III. Efficiency of Desiccants in the Drying<sup>a</sup> of Me<sub>2</sub>SO<sup>b</sup>**

desiccant	residual solvent water content, ppm		
	6 h	24 h	72 h
4A molecular sieves	978	471	332
3A molecular sieves	1050	448	269
none			
P <sub>2</sub> O <sub>5</sub>			
B <sub>2</sub> O <sub>3</sub>			
CaH <sub>2</sub>	1560		1820
BaO	1450	1330	1770
CaO	2060		1740
Al <sub>2</sub> O <sub>3</sub>	1840	1900	1920
K <sub>2</sub> CO <sub>3</sub>	2280	2200	
KOH (powdered)	2130 <sup>h</sup>		
CaSO <sub>4</sub>	2140		

<sup>a</sup> Static drying modes unless otherwise specified. <sup>b</sup> Desiccant loading 5% w/v; sequentially dried sample, 72 h. <sup>c</sup> Fractionally distilled sample. <sup>d</sup> Distilled sample. <sup>e</sup> C for 24 h followed by distillation. <sup>f</sup> Yellow colored solutions.

Agent	Capacity <sup>a</sup>	Speed <sup>b</sup>	Comments
CaSO <sub>4</sub>	1/2 H <sub>2</sub> O	Very fast (1)	Sold commercially as "Drierite" with or without a color indicator; very efficient. When dry the indicator (CoCl <sub>2</sub> ) is blue, but turns pink as it takes on H <sub>2</sub> O (capacity CoCl <sub>2</sub> ·6H <sub>2</sub> O); useful in temperature range -50° to +86°. Some organic solvent leach out, or change the color of CoCl <sub>2</sub> (acetone, alcohols, pyridine, etc.).
CaCl <sub>2</sub>	6 H <sub>2</sub> O	Very fast (2)	Not very efficient; use only for hydrocarbons and alkyl halides (forms solvates, complexes, or reacts with many N and O compounds).
MgSO <sub>4</sub>	7 H <sub>2</sub> O	Fast (4)	Excellent general agent; very inert but may be slightly acidic (avoid with very acid-sensitive compounds). May be soluble in some organic solvents.
Molecular Sieve 4A	High	Fast (30)	Very efficient; predrying with a more common agent recommended (see below for details on molecular sieves). Sieve 3A also excellent.
Na <sub>2</sub> SO <sub>4</sub>	10 H <sub>2</sub> O	Slow (290)	Very mild, inefficient, slow, inexpensive, high capacity; good for gross predrying, but do not warm the solution.
K <sub>2</sub> CO <sub>3</sub>	2 H <sub>2</sub> O	Fast	Good for esters, nitriles, ketones and especially alcohols; do not use with acidic compounds.
NaOH, KOH	Very high	Fast	Powerful, but used only with inert solutions in which agent is insoluble; especially good for amines.
H <sub>2</sub> SO <sub>4</sub>	Very high	Very fast	Very efficient, but use limited to saturated or aromatic hydrocarbons or halides (will remove olefins and other "basic" compounds).

**Table I. Desiccant Efficiency in the Drying<sup>a,b</sup> of a Pyridine<sup>c</sup> Series**

desiccant	residual water content, ppm			
	pyridine	2-methylpyridine	2,6-dimethylpyridine	2,4,6-trimethylpyridine
CaH <sub>2</sub>	39 (14) <sup>e</sup>	84	248 (138) <sup>e</sup>	132
CaC <sub>2</sub>	44 (10) <sup>e</sup>	71	519	8
BaO	101	27	360	33
4A sieves	106 (0.3) <sup>f</sup>		268 (126)	
3A sieves	117	55	200 (128)	47
benzene azeotrope	125	40	207	390
KOH powder	152	176	325	27
Na	388			
CaO	962		935	
silica gel	926			
Al <sub>2</sub> O <sub>3</sub>	1306			

<sup>a</sup> Static drying modes unless specified otherwise. <sup>b</sup> Water content assayed by the radioisotope technique. <sup>c</sup> Desiccant loading 5% w/v; initial water content 2500 ppm (0.25% w/w). <sup>d</sup> 24-h drying times unless specified otherwise. <sup>e</sup> 168-h drying time. <sup>f</sup> Sequentially dried sample, 24 h.

**Table I. Desiccant Efficiency in Drying<sup>a,b</sup> of Some Common Lower Alcohols<sup>c</sup>**

desiccant	residual water content, ppm			
	methanol <sup>d</sup>	ethanol <sup>e</sup>	2-butanol <sup>f</sup>	tert-butyl alcohol <sup>g</sup>
3A sieves (bead)	95	99	645 (9) <sup>h</sup>	428 (160) <sup>i</sup>
3A sieves (powder) <sup>j</sup>	940	18	14	13
MgI <sub>2</sub> <sup>k</sup>	97 (12) <sup>l</sup>	50 (53) <sup>m</sup>		
CaH <sub>2</sub>	125	99	17 <sup>n</sup>	406 (20) <sup>o</sup>
Na <sup>p</sup>		1800 <sup>n</sup>	2400 <sup>n</sup>	406 (10) <sup>o</sup>
Na/dicarboxylic acid ester <sup>q</sup>		92 <sup>n</sup>	36 <sup>n</sup>	
4A sieves (bead)	440	401		406
5A sieves (bead)	475	875		
CaC <sub>2</sub>	490	338 (199) <sup>r</sup>	409	430 <sup>n</sup> (662) <sup>o</sup>
BaO	1000			
Ca	1000			
K <sub>2</sub> CO <sub>3</sub>				860
CaO				750
KOH powder				770
ion exchange resin				640

<sup>a</sup> Static drying modes unless specified otherwise. <sup>b</sup> Water content assayed by the Karl Fischer method. <sup>c</sup> Desiccant loading 5% w/v with a drying period of 24 h unless specified otherwise. <sup>d</sup> Initial water content 1010 ppm. <sup>e</sup> Initial water content 1500 ppm. <sup>f</sup> Initial water content 1000 ppm. <sup>g</sup> Initial water content 1030 ppm. <sup>h</sup> 96-h drying period. <sup>i</sup> 168-h drying period. <sup>j</sup> Analysis was performed after settling of desiccant, 3-6 h. <sup>k</sup> Weight of magnesium in accord with general practice, i.e., 0.5% w/v. <sup>l</sup> See text. <sup>m</sup> Weight of magnesium 2% w/v. <sup>n</sup> Distilled sample. <sup>o</sup> Initial water content 1670 ppm, distilled sample. <sup>p</sup> Weight of sodium 3% w/v. See ref 32. <sup>q</sup> Ratio of sodium to 2-butyl succinate for 2-BuOH and to diethyl phthalate for ethanol in accord with general practice (see ref 7c), i.e., Na, 0.3 mol L<sup>-1</sup>; dicarboxylic acid ester, 0.14 mol L<sup>-1</sup>. <sup>r</sup> Stirred sample. <sup>s</sup> No apparent drying.

**III. Dependence of Drying Efficiency on Desiccant Loading in the Drying of Grossly Wet Diethyl Ether<sup>a</sup>**

desiccant	desiccant loading % w/v	residual solvent water content, ppm					capacity <sup>d</sup> % w/w
		5 min	15 min	30 min	60 min	360 min	
CaSO <sub>4</sub>	10 <sup>b</sup>		11400	9200	10200	10700	2.8-3.9
	20 <sup>b</sup>	6400	3800	2100			4.5
	20 <sup>c</sup>	9700	7500	5800			3.1
CaCl <sub>2</sub>	5 <sup>c</sup>			2100			19.6
	10 <sup>c</sup>		2400	2100	1900	850	10.1
	20 <sup>c</sup>		2100	1400	900		4.9

<sup>a</sup> Initial water content = 14700 ppm; drying temperature = 22 °C. Activation temperature: <sup>b</sup> = 220 °C. <sup>c</sup> = 350 °C. <sup>d</sup> Given by weight of water absorbed per unit of desiccant expressed as a percentage.