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Slush Baths

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The preparation of constant temperature slush baths is described and 86 common slush baths are tabulated in order of decreasing temperature from 13° to −160° C. Some experimental applications of these cooling agents are also described.

THE ACCOMPANYING TABLE lists 86 common solvents and their slush bath temperatures in order of decreasing temperature from 13° to −160° C. A slush bath can be defined as a coolant consisting of a low melting liquid which has been partially frozen by mixing with liquid nitrogen. It is prepared by slowly pouring liquid nitrogen into a Dewar flask containing the solvent while continuously stirring the mixture until the desired consistency is obtained. When properly mixed, the consistency of the crystallized solvent is that of a fluid slush which will maintain a constant temperature as long as the bath is kept slushy by occasionally blending in more liquid nitrogen.

Rondeau and Harrah (*2*) made use of an ethyl bromide slush bath in measuring the melting point of 3-hexyne. The temperature rise of this particular slush was approximately 0.2° C. per minute when left standing at room temperature in an uncovered 47 × 125 mm. Dewar flask.

With certain solvents, such as diethylene glycol and some of the alcohols, a heavy sirup is formed. Although not as convenient to handle, a viscous bath is still useful for cooling purposes. The solvents in Table I that form a highly viscous coolant have been marked with an asterisk.

The temperatures listed in the table are given to the nearest degree centigrade. Measurements were made with a calibrated toluene thermometer for temperatures above −95° C. and with a calibrated pentane thermometer for readings below −95° C. All of the solvents used were reagent grade chemicals. In general, the purer the compound, the narrower its slush bath temperature range; however, the variation in temperature with purity has not been studied.

Slush baths are especially useful in degassing liquids and fractionating mixtures. Newton (*1*) has designed a low temperature reflux condenser in which the liquid can be refluxed under vacuum at a temperature where its vapor pressure is negligible. The method requires the use of a constant temperature cooling bath to maintain the desired temperature. The convenience of a table of slush bath temperatures in using such a technique is readily apparent.

Volatile mixtures can be separated in a vacuum system by fractional condensation through a series of three traps cooled to successively lower temperature. Here again, the judicious selection of the proper slush bath temperature determines the efficiency of the separation.

Table I. Materials for Low Temperature Slush Baths (with Liquid Nitrogen)

Solvent	Temp., ° C.	Solvent	Temp., ° C.
p-Xylene	13 ±1°	Ethyl acetate	−84
p-Dioxane	12	*n*-Hexyl bromide	−85
Cyclohexane	6	Methyl ethyl ketone	−86
Benzene	5	Acrolein	−88
Formamide	2	Amyl bromide	−88
Aniline	−6	*n*-Butanol [a]	−89
Diethylene glycol [a]	−10	*s*-Butanol [a]	−89
Cycloheptane	−12	Isopropyl alcohol [a]	−89
Methyl benzoate	−12	Nitroethane	−90
Benzonitrile	−13	Heptane	−91
Benzyl alcohol	−15	*n*-Propyl acetate	−92
Propargyl alcohol	−17	2-Nitropropane	−93
1,2-Dichlorobenzene	−18	Cyclopentane	−93
Tetrachloroethylene	−22	Ethyl benzene	−94
Carbon tetrachloride	−23	Hexane	−94
1,3-Dichlorobenzene	−25	Toluene	−95
Nitromethane	−29	Cumene	−97
o-Xylene	−29	Methanol	−98
Bromobenzene	−30	Methyl acetate	−98
Iodobenzene	−31	Isobutyl acetate	−99
m-Toluidine	−32	Amyl chloride	−99
Thiophene	−38	Butyraldehyde	−99
Acetonitrile	−41	Propyl iodide	−101
Pyridine	−42	Butyl iodide	−103
Benzyl bromide	−43	Cyclohexene	−104
Cyclohexyl chloride	−44	*s*-Butyl amine	−105
Chlorobenzene	−45	Isooctane	−107
m-Xylene	−47	1-Nitropropane	−108
n-Butyl amine	−50	Ethyl iodide	−109
Benzyl acetate	−52	Propyl bromide	−110
n-Octane	−56	Carbon disulfide	−110
Chloroform	−63	Butyl bromide	−112
Methyl iodide	−66	Ethyl alcohol [a]	−116
tert-Butyl amine	−68	Isoamyl alcohol [a]	−117
Trichloroethylene	−73	Ethyl bromide	−119
Isopropyl acetate	−73	Propyl chloride	−123
o-Cymene	−74	Butyl chloride	−123
p-Cymene	−74	Acetaldehyde	−124
Butyl acetate	−77	Methyl cyclohexane	−126
Isoamyl acetate	−79	*n*-Propanol [a]	−127
Acrylonitrile	−82	*n*-Pentane	−131
n-Hexyl chloride	−83	1,5-Hexadiene	−141
Propyl amine	−83	*iso*-Pentane	−160

[a] High viscosity slush.

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