

MARSDEN
SOLVENTS
MANUAL

SOLVENTS

AND ALLIED SUBSTANCES

MANUAL

with

SOLUBILITY CHART

Compiled and Edited

by

C. MARSDEN

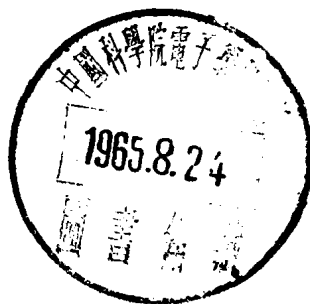
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P R E F A C E

DURING the past ten years or so the chemical industries have expanded at a rate altogether unprecedented, and this is especially true of those sections concerned with the manufacture of organic materials. In particular, the rapid evolution of a whole new industry based on petroleum has made available in bulk a wide range of solvents and allied products which until a few years ago belonged to the category of fine chemicals rather than of industrial materials.

So rapid has this growth been and so prolific the outpouring of technical and commercial information which has accompanied it that inevitably documentation has fallen behind and the need for new and up-to-date books of reference is pressing. The difficulties of publishing during the war years greatly aggravated the position.

In the course of his professional work the writer has necessarily accumulated a mass of information on solvents and allied chemicals currently in commercial production. This has been culled from a great number of sources both academic and commercial. In the course of coordinating it extraordinarily wide divergencies have been noted in published data, attributable often to the use of early commercial material of uncertain purity, often to the lack of standardisation of apparatus and methods used in making measurements.

A careful and critical study of this conflicting data has been made and the outcome is offered in this volume. That all the information contained in it should be completely accurate is too much to hope, but the more serious discrepancies and variations in published information and figures have been eliminated by careful selection in an effort to ensure that the data here presented is as accurate as possible in terms of present knowledge.

So vast is the bulk of information to be sifted and so many the gaps in published information that this first edition cannot possibly be as complete and comprehensive as might be wished. The author will welcome all cooperation from users of the book in the way of correction or fresh data for the improvement of subsequent editions.

C. MARSDEN.

London, April 1954.

ACKNOWLEDGMENTS

The writer would like to express his sincere thanks to the Directors of
British Industrial Solvents

for their kindness in allowing him to publish this volume and to those colleagues and friends who have helped in the often tedious task of compiling and editing it by reading and correcting the manuscript and by their constructive and helpful criticism. In this connection his special thanks are due to Mr. P. J. Edmonds of the Cleaver-Hume Press for invaluable assistance in the matter of presentation, and to Mr. J. A. Oates whose skill and thoroughness so greatly eased the burden of proofing.

He is deeply indebted also to the many companies mentioned throughout the text who have so generously furnished much valuable and up-to-date information, much of it compiled by their own laboratories, and have graciously permitted its reproduction.

British Standard Specifications are obtainable from
The British Standards Institution.

A.S.T.M. Specifications may be obtained from
The American Society for Testing Materials.

DEFINITION OF TERMS

Boiling Point

The boiling point of a liquid is that temperature at which its vapour pressure becomes equal to the pressure of the surrounding atmosphere. In general the boiling point is quoted for a pressure of one standard atmosphere, unless some other pressure is specifically stated.

1 Standard atmosphere = 760 mm Hg = 14.696 p.s.i.a.

Melting Point

The melting point of a pure substance is that temperature at which the solid substance is in equilibrium with liquid. In a slightly impure material such an equilibrium may be maintained over a range of temperatures and in such a case the term melting point should be applied to the lowest temperature at which liquid and solid coexist, that is at which the solid substance first begins to melt. The upper temperature at which solid particles first appear in the liquid is more correctly termed 'freezing point'. In the literature and in manufacturers' technical information these two terms are frequently confused, and it is often impossible to decide which temperature is meant.

In the monographs which follow, therefore, the term 'melting point' has been used throughout to imply that temperature at which the substance is largely but not necessarily completely solidified.

Flash Point

The flash point of a solvent is the lowest temperature at which the vapour concentration immediately above the liquid surface is high enough to form an explosive mixture with air, so that a spark or flame will ignite it and possibly fire the liquid also. This temperature varies with atmospheric pressure and the values in the monographs are for a standard atmospheric pressure of 760 mm Hg or 14.696 p.s.i.a.

The flash point varies also according to the form of apparatus in which it is measured. Several different forms of apparatus are in common use but these fall basically into two types, 'open cup' and 'closed cup'. Broadly the 'open cup' flash point is from 5° to 15°F higher than the 'closed cup' value, but the difference varies from solvent to solvent.

In Britain the Abel and the Pensky-Martin apparatus are commonly used; in the United States the usual apparatus is either the Tag or Cleveland. All these can be used to determine either open or closed cup flash-points.

Fire Point

This is the lowest temperature at which the liquid will ignite and continue to burn when open to air. It is usually somewhat higher than the open cup flash point.

Auto-ignition Temperature

This is defined as the temperature at which the solvent in contact with air will ignite without the presence of a spark or flame. If liquid is spilled on hot metal at this temperature or higher it will catch fire spontaneously. The auto-ignition temperature is usually fairly high and difficult to measure with precision; for this reason a temperature range is usually quoted.

Specific Gravity

The specific gravity of a liquid is the ratio between the weight of a given volume of the liquid and that of an equal volume of water. The temperatures of both liquid and water must be stipulated. Usually both liquid and water are weighed at the same temperature but sometimes the weight of the water is taken at 4°C. Since at this temperature 1 millilitre of water weighs 1 gram precisely, the specific gravity under these conditions is equal to the weight per millilitre of the liquid at the temperature stated.

Refractive Index

All refractive indices quoted in the following monographs are for the sodium D line at the temperatures stipulated.

Explosive Limits in Air

For each inflammable substance there is a range of vapour concentrations in air within which a flame will propagate or an explosion occur if the mixture is ignited by spark or flame. This range is defined by the upper and lower explosive limits respectively for the material in question. Values quoted in the monographs are for 20°C except where otherwise stated.

At higher or lower vapour concentrations there is either insufficient vapour or too little air to sustain and propagate a flame.

The explosive limits vary with atmospheric pressure and quoted values are for one standard atmosphere of 760 mm Hg.

Coefficient of Cubic Expansion

This is the fractional increase in volume which takes place when the temperature of a substance is changed by 1 degree. Values in the monographs are for a temperature change of 1°C. The value per degree Fahrenheit is of course five-ninths of this.

Specific Heat

The specific heat of a substance is the ratio of its thermal capacity to that of water at the same temperature. It is equal to the number of calories required to raise the temperature of the liquid by 1°C and varies with the temperature at which it is determined.

Latent Heat of Evaporation

This is the quantity of heat required to convert unit weight of the substance from liquid to vapour at the boiling point. It varies with atmospheric pressure. Values quoted in the monographs are for a standard atmosphere of 760 mm Hg.

Latent Heat of Fusion

This is the quantity of heat required to convert unit weight of the substance from solid to liquid at the melting point. It varies with pressure, though not greatly. Values in the following monographs are for a pressure of one standard atmosphere of 760 mm Hg.

Electrical Conductivity

This is the reciprocal of the volume resistivity or specific resistance. Volume resistivity is defined as the resistance in ohms of a cube of 1 centimetre, measured between opposite faces. It varies with temperature.

Dielectric Constant

The dielectric constant of a substance is the ratio between the capacity of a condenser with the substance as dielectric and that of a similar condenser with a vacuum as its dielectric.

Critical Temperature

This is the temperature above which a vapour or gas cannot be liquefied by pressure.

Critical Pressure

This is defined as the pressure at which the vapour of a substance exists in equilibrium with liquid at the critical temperature.

Critical Density

The critical density is the vapour density of a substance at critical temperature and critical pressure.

Dilution Ratio

When a solution of cellulose nitrate in an active solvent is diluted with a non-solvent a critical point is reached after which further addition of non-solvent causes gelling or precipitation of the cellulose nitrate. The ratio of non-solvent to solvent at this point is termed the 'dilution ratio'.

Values quoted in the monographs are for '½-second' cellulose nitrate and are shown for different diluents. Where 'petroleum naphtha' is mentioned as the diluent this refers to aliphatic hydrocarbon such as 'Special Boiling Point' petroleum spirit No. 3 or a similar fraction.

Azeotropes

An azeotrope is a mixture of two or three liquids having a constant boiling point so that on distillation the mixture comes over unchanged in composition. A binary azeotrope may boil at a temperature below that of the most volatile component or above that of the less volatile.

Ternary azeotropes usually boil at a lower temperature than any of the three ingredients alone.

The composition of an azeotrope depends on atmospheric pressure, as does its boiling point. Data in the monographs refers to a pressure of one standard atmosphere of 760 mm Hg or 14.696 p.s.i.a.

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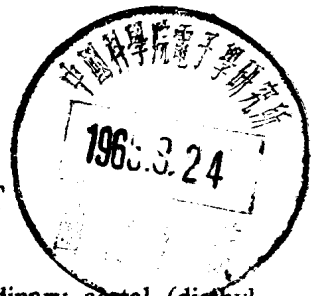
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ACETAL SOLVENT

Physical Characteristics and Properties

Acetal solvent, which should not be confused with ordinary acetal (diethyl acetal) is the azeotropic mixture of diethyl acetal and ethanol. It is sold on the British market for solvent purposes and is a much more active solvent than either diethyl acetal or ethanol separately. It complies with the following specification.

Distillation range	90% 75°-85°C
Flash point	38°F (3°C) approx
Specific gravity at 15.5°/15.5°C	0.82
Acidity (as acetic acid)	0.01% (max)

Its properties other than those indicated by the specification are intermediate between the properties of diethyl acetal and ethanol.

Manufacturers

Howards of Ilford Limited

ACETONE



(Dimethyl ketone, propanone-2)

Physical Characteristics and Properties

A colourless mobile liquid, somewhat hygroscopic, with the rather mint-like odour of the simpler aliphatic ketones.

Molecular weight	58.08
Boiling point at 760 mm Hg	56.2°C
Melting point	-94.3°C
Flash point (closed cup)	1°F (-17°C)
(open cup)	15°F (-9°C)
Auto-ignition temperature	600°-650°C
Specific gravity	See table below
Refractive index at 20°C	1.3591
at 25°C	1.3573
Calorific value	7373 cal/g
Specific heat at 20°C	0.528 cal/g
Coefficient of cubic expansion at 20°C	0.00143
mean from 0°-100°C	0.00162
Electrical conductivity at 20°C	5.5×10^{-8} recip. ohms
Dielectric constant at 20°C	21.45

Explosive mixtures with air at 20°C:	
upper limit	13.0 vols per cent
lower limit	2.15 vols per cent
Latent heat of evaporation	125.3 cal/g
Latent heat of fusion	23.4 cal/g
Evaporation rate	
(ether = 1)	2.1
(<i>n</i> -butyl acetate = 100)	72
Dilution ratios	
Water	0.21
Toluene	4.5
Xylene	3.7
Petroleum naphtha	0.6
Butanol	7.0
Viscosity	See table below
Surface tension at 20°C	23.7 dynes/cm
at 30°C	22.01 dynes/cm
Vapour pressure	See table below
Solubility in water	Miscible
Critical temperature	235°C
Critical pressure	47 atmospheres
Critical density	0.268 g/cm

SPECIFIC GRAVITY OF ACETONE

Temperature <i>T</i> °C	Sp. gr. at <i>T</i> °/20°C	Temperature <i>T</i> °C	Sp. gr. at <i>T</i> °/20°C
0	0.8141	30	0.7796
5	0.8081	35	0.7736
10	0.8026	40	0.7681
15	0.7966	45	0.7621
20	0.7911	50	0.7566
25	0.7851		

The effect of water up to about 10 per cent is to raise the specific gravity of acetone at the rate of approximately 0.0030 per 1 per cent by weight of water.

SPECIFIC GRAVITY OF ACETONE-WATER MIXTURES

Acetone content per cent w/w	Sp. gr. at 20°/20°C	Acetone content per cent w/w	Sp. gr. at 20°/20°C
10	0.985	60	0.895
20	0.972	70	0.872
30	0.955	80	0.847
40	0.938	90	0.850
50	0.917	100	0.791

ACETONE

VAPOUR PRESSURE OF ACETONE

<i>Temperature</i> °C	<i>Vapour pressure</i> mm Hg	<i>Temperature</i> °C	<i>Vapour pressure</i> mm Hg
-94.8	0.017	20	184.8
-90	0.021	25	229.2
-70	0.34	30	282.7
-50	2.4	35	346.4
-30	11.2	40	421.5
-10	38.7	45	510.5
-2	60.0	50	612.6
+5	89.1	56.2	760.0
10	115.6		

<i>Temperature</i> °C	<i>Vapour pressure in atmospheres</i>	<i>Temperature</i> °C	<i>Vapour pressure in atmospheres</i>
60	1.14	100	3.67
70	1.58	120	6.01
80	2.12	150	11.5
90	2.81	200	28.0

VISCOSITY OF ACETONE

<i>Temperature</i> °C	<i>Viscosity in centipoises</i>	<i>Temperature</i> °C	<i>Viscosity in centipoises</i>
-92.5	2.148	-10.0	0.450
-80	1.487	0	0.399
-59.6	0.932	15	0.337
-42.5	0.695	25	0.316
-30.0	0.575	30	0.295
-20.9	0.510	41	0.280
-13.0	0.470		

VISCOSITY OF ACETONE-WATER MIXTURES AT 25°C

<i>Acetone per cent w/w</i>	<i>Viscosity in centipoises</i>	<i>Acetone per cent w/w</i>	<i>Viscosity in centipoises</i>
100	0.316	50	1.25
90	0.44	40	1.35
80	0.60	30	1.35
70	0.83	20	1.26
60	1.05	10	1.09

Specifications	Typical British Commercial	Typical American Commercial	B.S.S. 509, 1950	A.S.T.M. D239-33
Specific gravity at 15.5°/15.5°C 20°/20°C	0.796-0.798 0.791-0.793	— 0.7915-0.7935	0.796-0.798 0.791-0.793	— 0.791-0.799
Distillation range	98% between 55.5°-56.5°C	100% within 1°C including 56.1°C	95% between 55.5°-56.5°C	100% between 55°-57.5°C
Residue Miscibility with water Water content	0.001% (max) Complete Miscible with 10 vols kerosine	0.001% (max) Complete Miscible with 19 vols. gasoline	0.005% (max) Complete Miscible with 19 vols. CS ₂	0.005% (max) Complete Miscible with gasoline in all proportions
Acidity	0.002% (max) as acetic acid	0.002% (max) as CO ₂	0.002% (max) as acetic acid	0.002% (max) as CO ₂
Alkalinity	None to methyl red	None to <i>p</i> -nitrophenol	None to methyl red	None to <i>p</i> -nitrophenol
Alcohol content	10 minutes	5 minutes	—	—
Permanganate test	2 hours (min)	2 hours (min)	30 minutes (min)	30 minutes (min)
Purity	—	99.5%	—	98% (min)
Colour	Water-white	10 APHA units (max)	Colourless	Water-white

Source and Manufacture

Formerly obtained almost exclusively from the products of destructive distillation of wood, acetone was later produced commercially by the catalytic conversion of either ethanol or acetic acid in the vapour phase. More recently these methods have been superseded by catalytic dehydrogenation of isopropanol, by which process most British and American acetone is now manufactured.

It is also made on the commercial scale by the Fernbach-Strange-Weizmann fermentation process which produces butanol simultaneously. There is no significant difference in quality between the finished products from these various processes.

Industrial Grades

There is in effect only one quality of acetone in commercial production nowadays, although material from different processes and different manufacturers differs slightly in the nature and quantity of trace impurities present. British and American makers' current specifications show only trivial differences and those quoted on page 4 are typical. Some official specifications are also included for reference.

Azeotropes

BINARY MIXTURES

<i>Acetone per cent w/w</i>	<i>Second component</i>	<i>per cent w/w</i>	<i>Boiling point °C</i>
88.5	Carbon tetrachloride	11.5	56.28
33	Carbon disulphide	67	39.25
20.5	Chloroform	79.5	64.5
13	Methyl iodide	87	41.5
88	Methanol	12	56.4
30	1: 1-dichlorethane	70	57.55
87	Chloromethyl methyl ether	13	56.1
65	Ethyl iodide	35	55.5
48	Methyl acetate	52	55.6
99	1-Bromopropane	1	56.33
50	2-Bromopropane	50	54.0
8	<i>n</i> -Propyl nitrite	92	47.5
67	1-Propane-thiol	33	54.5
82.5	Trimethyl borate	17.5	55.55
20	<i>n</i> -Propylamine	80	48.5
85	<i>iso</i> -Butylamine	15	56.0
38	Diethylamine	62	51.5
20	<i>n</i> -Pentane	80	32.0
90	Ethyl propyl ether	10	56.1
47	1: 5-Hexadiene	53	47.5
59	<i>n</i> -Hexane	41	49.8

The above figures are drawn from the list compiled by L. H. Horsley and published in August 1947. The boiling point of pure acetone is shown as

56·35°C which is higher than the value given under "Properties" on page 1. It is probable therefore that the boiling points of those azeotropes in which acetone is largely predominant and the boiling point of which is given above as 56° or higher, are too high by 0·10–0·15°C.

TERNARY MIXTURE

Acetone forms a ternary mixture boiling at 38·04°C containing 23·98 per cent acetone, 75·21 per cent carbon disulphide and 0·81 per cent water.

Physiological Properties

Although acetone is one of the most widely used industrial solvents there are no recorded cases of fatal or even serious poisoning. Where excessive exposure to vapour has caused illness, recovery has been rapid and complete without detectable after-effects.

At levels approaching 600 parts per million which is the accepted maximum concentration for moderately long exposure, acetone vapour is quite irritating to eyes, nose and throat, although it may not be sufficiently so to be really unpleasant, especially to workers who have become habituated. It has a slight narcotic action and may produce headache and slight nausea. With some subjects these symptoms may appear at levels around 400 parts per million but they are rapidly alleviated on removal from exposure and there are no apparent sequelæ. Practically no data is available on the effects of mixed vapours, but there have been suggestions that to allow for synergesis, it may be desirable with mixed solvents to keep the level of acetone vapour below that which would be acceptable with acetone alone.

DETERMINATION OF ACETONE VAPOUR IN THE ATMOSPHERE

The best method is that of Jacobs. A large sample taken in a partially evacuated flask is shaken with a few millilitres of 0·1N iodine solution and the excess iodine titrated with thiosulphate, standardized against high-quality commercial acetone 1 ml 0·1N iodine solution has been found equivalent to 0·967 mg acetone. The method is very sensitive but subject to interference by other substances which react with iodine.

Storage and Handling

Not appreciably corrosive to metals. Iron, mild steel, copper or aluminium are suitable for plant and containers, but in the case of old or recovered acetone which may have developed appreciable acidity, copper may cause slight discoloration and suffer perceptible corrosion.

Fire Hazard. Highly inflammable and the vapour forms explosive mixtures with air (see page 2).

Regulations. The Cellulose Solutions Regulations govern the use of acetone in lacquers etc. and there may be local by-laws affecting storage and use.

Special regulations are in force covering transport by road, rail and water.

Manufacturers

British Celanese Limited

British Industrial Solvents

Commercial Solvents (Great Britain) Limited