

## AN APPARATUS FOR THE MEASUREMENT OF MAXIMUM EXPERIMENTAL SAFE GAPS AT STANDARD AND ELEVATED TEMPERATURES

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### Summary

The paper describes an apparatus developed for determining Maximum Experimental Safe Gaps (MESGs) of gases and vapours up to 130°C. The apparatus is particularly useful for liquids that vaporise only at elevated temperatures. The method is rapid and accurate. The MESGs of over sixty organic compounds are listed.

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### Introduction

The Maximum Experimental Safe Gap (MESG) of any gas or vapour that can form a flammable atmosphere with air is measured as the maximum normal distance between parallel surfaces that will prevent the explosion of any gas—air or vapour—air mixture on one side of the gap from igniting any mixture of the same gases on the other side. The MESG is important for the design of flameproof enclosures for electrical equipment used in flammable atmospheres.

Although a theory developed by Phillips [1] explains the behaviour of the safe gap, and gives a method for MESG calculation, in many cases the data needed to carry out the calculation are either unreliable or not available, so it is still necessary to rely on experimental measurement when practical values of the MESG are required.

This paper describes an apparatus developed for the routine measurement of MESGs by the Research and Laboratories Services Division (RLSD) of the Health and Safety Executive. The main feature of the apparatus is that measurements can be obtained for liquids that form flammable vapours only at elevated temperatures.

### Apparatus

An apparatus for the determination of MESGs consists of a chamber connected by a variable gap to a much larger surrounding chamber. The gas or vapour being tested is mixed with air to form an explosive mixture which

is ignited in the internal chamber by an electric spark. The external chamber contains an explosive mixture of the same fuel and air, but not necessarily with the same composition as the internal mixture.

The connecting gap is varied by small standard amounts until, for a given internal fuel-air mixture, the safe gap that just prevents ignition of any external mixture is found. The concentration of the internal fuel-air mixture that has the smallest safe gap must be found by trial and error, and the smallest safe gap is taken as the MESG of that particular fuel. In practice the MESG is obtained on the understanding that a specified probability of external ignition is not exceeded. A gap slightly larger than the MESG allows a greater probability of external ignition for at least one combination of fuel-air mixtures.

Over many years a large number of MESGs have been measured using a spherical 8-litre chamber connected to a surrounding chamber by an equatorial flange gap 25 mm across [2]; internal ignition is from a spark gap positioned 10 mm from the internal edge of the flange. The use of a more compact apparatus has been recommended by the International Electrotechnical Commission (IEC) [3] and the RLSD apparatus follows the IEC guidelines, but not the details; the design is a modification of an apparatus reported by Physikalisch-Technische Bundesanstalt (PTB) [4]. The RLSD apparatus is shown diagrammatically in Fig. 1.

- A... Flange gap - 25 mm flame path
- B... Spark gap - 3 mm gap positioned 14 mm from internal edge of flange gap.
- C... Volume of internal chamber - 20 ml
- D... Volume of external chamber - 4 litres
- E... Self-sealing injection - port for liquids (hypodermic injection)
- F... Tap for air-purging
- G... Vernier screw
- H... Fan motor
- I... Hand pump for circulating mixture
- J... Glycerol bath
- K... Stirring fan
- L... Heating element
- M... Entry ports

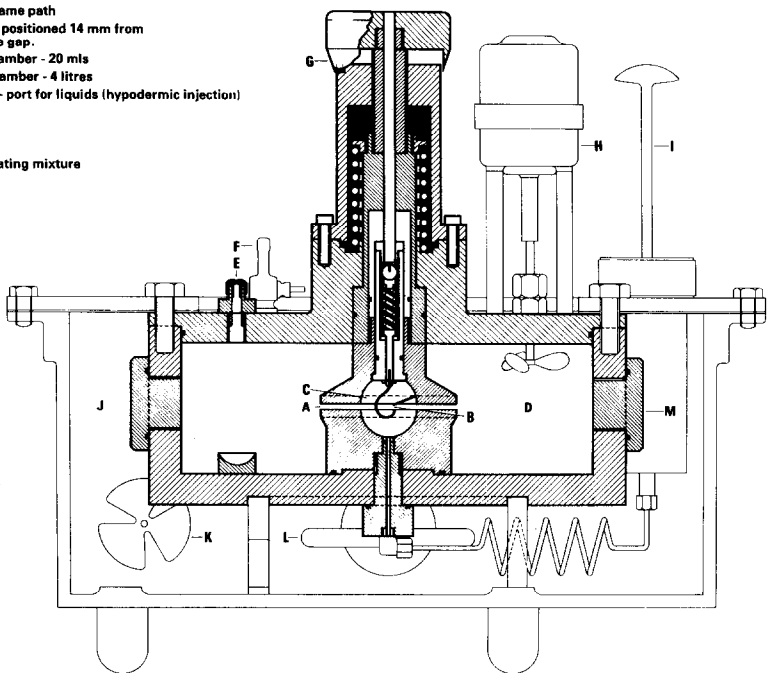


Fig. 1. RLSD apparatus for measuring MESGs at normal and elevated temperatures.

## *Description of apparatus*

### *Internal chamber*

The features of the internal chamber of the RLSD apparatus are similar to those of the PTB apparatus. The chamber is a sphere of 17 mm radius, with a volume of approximately 20 ml and is made of stainless steel. (A brass chamber has been used in some experiments with non-corrosive compounds in a separate apparatus used only for room temperature measurements.)

### *External chamber*

The external chamber is a 4.5 litre cylinder of diameter 255 mm and height 90 mm, and is somewhat larger than the PTB design. The external edge of the flange gap is unobstructed as the presence of obstacles can lead to erroneous results [5]. The external chamber contains a stirring fan, and there are four entry ports in the wall of the chamber which allow for alignment of gap.

### *Flange gap*

The adjustable flange gap lies in the equatorial plane of the internal chamber and has a flame path of 25 mm. The external edges of the flange gap are bounded by 5-mm-wide flat faces and the corners of the gap are sharp, conforming to the PTB design. The gap is adjusted by a screw-thread arrangement and the exact width of the gap is measured from a scale on the micrometer head.

### *Spark ignition*

The mixture inside the internal chamber is ignited by means of a spark across a 3-mm wide vertical spark gap. The spark gap is positioned in the equatorial plane 14 mm from the internal edge of the flange gap. Experiments have shown this position to be the most effective ignition point for this kind of apparatus [4].

### *Method of heating the apparatus*

The apparatus is enclosed in a glycerol bath. The bath is heated by an electric element and the glycerol is mixed and circulated by a stirring fan.

### *Introduction of gas or liquid*

Phillips [1] has shown that there is no need to differentiate between the most incendive internal and the most easily ignitable external fuel-air mixtures. The fuel-air mixture can be the same at each side of the flange gap and a single most dangerous mixture found at which the MESG can be measured.

If the test substance is a gas, a fuel-air mixture of known proportions is circulated through the apparatus. If the test substance is a liquid, a known amount of liquid is injected into the apparatus through a septum seal. Mixing and circulation inside the external chamber is by a stirring fan; a single

stroke hand pump which is immersed in the glycerol bath is used to circulate the gas or vapour through the internal chamber. A uniform mixture can therefore be obtained in both chambers without a risk of vapour condensing in the circulation system.

### Procedure

The compactness of this apparatus, and a fuller knowledge of the behaviour of the MESH, allow simplification of the experimental procedure, offering several advantages over the 8-litre and the PTB apparatus:

- (1) A smaller amount of fuel is required for a complete measurement.
- (2) The small size of the internal chamber compared to the external chamber allows, without introducing appreciable error, four internal ignitions to be made before fresh fuel need be introduced, provided there is no external ignition and the mixture is well circulated after each test.
- (3) Phillips and Shaw [6] have shown that a useful practical definition of the MESH is obtained if the basis for the MESH measurement is specified such that a gap wider than the MESH will result in at least one external ignition during twenty consecutive internal ignitions for at least one fuel-air mixture. In twenty tests with the gap equal to the MESH, there will be no external ignitions.
- (4) A distinct advantage of a smaller apparatus is that it can be more readily designed for measurements at elevated temperatures. The glycerol bath allows measurements up to 130°C. For those liquid fuels which require the apparatus to be heated, the MESH must be measured at a temperature that will give a vapour concentration corresponding to the most dangerous mixture of the fuel, which usually lies on the fuel-rich side of the stoichiometric mixture. The MESHs were measured at a temperature 10°C greater than the temperature that would give a vapour concentration corresponding to a fuel-air mixture of equivalence ratio 1.5. Part of the temperature increase provides a margin of safety preventing condensation of vapour within the apparatus. The vapour pressure data used in the calculations were obtained from Jordan [7]. The temperature is held constant throughout the testing of a fuel. The measurement of the MESH begins once the bath has reached the required temperature and has been held at that temperature for some time to allow the whole apparatus to reach the required temperature.
- (5) An MESH can be measured in days with the RLSD apparatus rather than the lengthy period needed for the statistical analysis of the 8-litre apparatus or for the procedure of the PTB apparatus. The MESHs are in close agreement with those from the 8-litre and PTB apparatus.

### *Preliminary estimate of the most dangerous mixture*

When the apparatus reaches the required temperature, a rapid search is made for a preliminary value of the most dangerous fuel-air mixture. The approximate safe gaps of a series of fuel-air mixtures are measured roughly as the gaps that will allow at least one external ignition in four internal ignitions. The width of the gap is increased or decreased in standard amounts of 0.02 mm and the mixture with the narrowest safe gap is taken as the preliminary estimate of the most dangerous mixture.

### *Determination of MESG*

The test begins by using the preliminary estimate of the most dangerous mixture to find a more accurate safe gap based on the twenty internal ignitions specification. The width of the safe gap is increased or decreased in standard amounts of 0.02 mm. The fuel concentration in the mixture is altered by a small amount (e.g. 0.05 ml of liquid; 0.25% of gas) and the safe gap for this mixture is determined. This is repeated until a mixture is found with a safe gap narrower than, or as narrow as, the safe gaps of the mixtures with slightly lower and slightly higher fuel concentrations. This value is taken as the MESG of the fuel at the temperature of the test. The ambient pressure is measured.

### **Corrections to MESG**

Previous routine measurements were seldom made at temperatures higher than 50°C, and results had been grouped on the assumption that they were obtained at room conditions. However, the MESG of a fuel decreases as temperature increases and the present results have been corrected to 20°C and 100 kPa (1 atm), so that MESGs can be compared under similar conditions.

#### *Pressure correction*

The results have been corrected to 100 kPa (1 atm) using a relationship between MESG and ambient pressure derived from measurements obtained in an automatic safe-gap apparatus [6].

The correction is:

$$\text{Corrected MESG (100 kPa)} = \frac{\text{Measured MESG}}{0.35 + (0.0065 \times P)}$$





where  $P$  is the ambient pressure in kPa.

#### *Temperature correction*

MESGs corrected to 100 kPa are subsequently corrected to 20°C. The correction is derived from results by PTB [4] of the effect of temperature on the MESGs of methane and ethylene. The temperature correction is not a simple function: it is dependent on the experimental temperature and the MESG at room temperature. The correction derived from PTB's methane results is different from that derived from their ethylene results. However,

TABLE 1

Experimental values of the maximum experimental safe gap  
(the compounds are listed under the heading of their organic type)

Name	Formula	Flash point (°C)	MESG (mm)	Temp (°C)	Corrected MESG (mm) (100 kPa, 20°C)	Group
<b>1. Alkenes</b>						
Propene	C=C-C	-	0.94	20	0.94	IIA
1-Butene	C=C-C-C	-80	0.94	20	0.94	IIA
2-Butene	C-C=C-C	-	0.89	20	0.89	IIB
1-Heptene	C=C-C-C-C-C-C	-1	0.97	20	0.97	IIA
<b>2. Alkynes</b>						
Propyne	C-C≡C	-	0.74	30	0.74	IIB
1-Butyne	C-C-C≡C	-	0.71	20	0.71	IIB
<b>3. Cyclic hydrocarbons</b>						
Cyclopropane		-	0.91	20	0.91	IIA
Cyclopentane		<-7	1.01	20	1.01	IIA
4,7 Methanoindene, 3a,4,7,7a-tetrahydro (Dicyclopentadiene)		32	0.87	70	0.91	IIA
<i>p</i> -Mentha-1,8-diene (Dipentene)		50	1.14	68	1.18	IIA
<b>4. Alcohols</b>						
1-Propanol	C-C-C-OH	15	0.86	51	0.89	IIB
1-Butanol	C-C-C-C-OH	29	0.88	64	0.91	IIA
1-Heptanol	C-C-C-C-C-C-C-OH	70	0.88	100	0.94	IIA
1-Octanol	C-C-C-C-C-C-C-C-OH	81	0.95	116	1.03	IIA
Allyl alcohol	C=C-C-OH	21	0.84	30	0.84	IIB

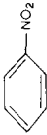

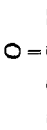
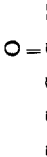
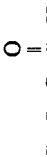
5. Nitro- compounds	Nitromethane	C-NO <sub>2</sub>	35	1.11	87	1.17	IIA
	Nitroethane	C-C-NO <sub>2</sub>	27	0.84	67	0.87	IIB
	1-Nitropropane	C-C-C-NO <sub>2</sub>	48	0.81	71	0.84	IIB
	Nitrobenzene		88	0.86	127	0.94	IIA
6. Chloro- hydrocarbons	Propene, 3-chloro- (Allyl chloride)	C=C-C-Cl	-31	1.17	20	1.17	IIA
	7. Amines	Propylamine	C-C-C-NH <sub>2</sub>	-	1.13	20	1.13
Butylamine		C-C-C-C-NH <sub>2</sub>	7	1.16	20	1.16	IIA
1,2-Ethanediamine		NH <sub>2</sub> -C-C-NH <sub>2</sub>	61	1.47	61	1.55	IIA
8. Thiols	Ethanethiol	C-C-SH	26	0.90	30	0.90	IIA
9. Aldehydes	Formaldehyde	H <sub>2</sub> C=O	-	0.57	20	0.57	IIB
	Acetaldehyde		-43	0.92	20	0.92	IIA
	Propionaldehyde		-10	0.84	20	0.84	IIB
	Butyraldehyde		-7	0.92	20	0.92	IIA
	Crotonaldehyde, (E)		12	0.81	20	0.81	IIA

TABLE 1 (continued)

Name	Formula	Flash point (°C)	MESG (mm)	Temp (°C)	Corrected MESG (mm) (100 kPa, 20°C)	Group
<b>10. Ketones</b>						
Acetone	$\begin{array}{c} \text{O} \\ \parallel \\ \text{C}-\text{C}-\text{C} \end{array}$	-18	1.04	20	1.04	IIA
2-Butane	$\begin{array}{c} \text{O} \\ \parallel \\ \text{C}-\text{C}-\text{C}-\text{C} \end{array}$	-5	0.92	20	0.92	IIA
2-Pentanone	$\begin{array}{c} \text{O} \\ \parallel \\ \text{C}-\text{C}-\text{C}-\text{C}-\text{C} \end{array}$	22	0.99	30	0.99	IIA
3-Pentanone	$\begin{array}{c} \text{O} \\ \parallel \\ \text{C}-\text{C}-\text{C}-\text{C}-\text{C} \end{array}$	13	0.88	48	0.90	IIA
<b>11. Carboxylic acids</b>						
Formic acid	$\begin{array}{c} \text{O} \\ \parallel \\ \text{HC}-\text{OH} \end{array}$	69	1.76	85	1.86	IIA
Acetic acid	$\begin{array}{c} \text{O} \\ \parallel \\ \text{C}-\text{C}-\text{OH} \end{array}$	40	1.69	73	1.76	IIA
<b>12. Ethers</b>						
Diethyl ether	$\text{C}-\text{C}-\text{O}-\text{C}-\text{C}$	-40	0.87	20	0.87	IIB
Dibutyl ether	$\text{C}-\text{C}-\text{C}-\text{C}-\text{O}-\text{C}-\text{C}-\text{C}-\text{C}$	25	0.86	50	0.88	IIB
2-Methoxyethanol	$\text{C}-\text{O}-\text{C}-\text{C}-\text{OH}$	51	0.81	63	0.84	IIB
2-Ethoxyethanol	$\text{C}-\text{C}-\text{O}-\text{C}-\text{C}-\text{OH}$	43	0.78	78	0.84	IIB
Ethanol, 2-(2 ethoxyethoxy)	$\text{C}-\text{C}-\text{O}-\text{C}-\text{C}-\text{O}-\text{C}-\text{C}-\text{OH}$	85	0.86	120	0.94	IIA
Ethanol, 2-(2 butoxyethoxy)	$\text{C}-\text{C}-\text{C}-\text{C}-\text{O}-\text{C}-\text{C}-\text{O}-\text{C}-\text{C}-\text{OH}$	100	1.01	133	1.11	IIA
Chlormethoxymethane	$\text{C}-\text{O}-\text{C}-\text{Cl}$	--	1.00	20	1.00	IIA



13. Esters								
Formic acid, methyl ester	$\text{HC} \begin{array}{c} \text{O} \\ \parallel \\ \text{O}-\text{C} \end{array}$	-32	0.94	20	0.94	20	0.94	IIA
Formic acid, ethyl ester	$\text{HC} \begin{array}{c} \text{O} \\ \parallel \\ \text{O}-\text{C}-\text{C}$	-20	0.94	20	0.94	20	0.94	IIA
Acetic acid, vinyl ester	$\text{C} \begin{array}{c} \text{O} \\ \parallel \\ \text{O}-\text{C}-\text{O}-\text{C}=\text{C} \end{array}$	-3	0.85	20	0.85	20	0.85	IIB
Acrylic acid, ethyl ester	$\text{C}=\text{C} \begin{array}{c} \text{O} \\ \parallel \\ \text{C}-\text{O}-\text{C}-\text{C} \\ \text{C} \text{ O} \end{array}$	-	0.86	20	0.86	20	0.86	IIB
Methacrylic acid, methyl ester	$\text{C}=\text{C} \begin{array}{c} \text{O} \\ \parallel \\ \text{C}-\text{O}-\text{C} \\ \text{C} \text{ O} \end{array}$	10	0.99	30	0.99	30	0.99	IIA
Methacrylic acid, ethyl ester	$\text{C}=\text{C} \begin{array}{c} \text{O} \text{ O} \\ \parallel \parallel \\ \text{C}-\text{C}-\text{O}-\text{C}-\text{C} \\ \text{O} \end{array}$	20	1.01	30	1.01	30	1.01	IIA
Acetoacetic acid, methyl ester	$\text{C}-\text{C} \begin{array}{c} \text{O} \\ \parallel \\ \text{O}-\text{C}-\text{O}-\text{C} \\ \text{O} \end{array}$	82	0.96	101	1.03	101	1.03	IIA
Acetoacetic acid, ethyl ester	$\text{C}-\text{C} \begin{array}{c} \text{O} \\ \parallel \\ \text{O}-\text{C}-\text{O}-\text{C}-\text{C} \\ \text{O} \end{array}$	85	0.98	103	1.04	103	1.04	IIA
Ethanol, 2-ethoxy-, acetate	$\text{C}-\text{C} \begin{array}{c} \text{O} \\ \parallel \\ \text{O}-\text{C}-\text{O}-\text{C}-\text{O}-\text{C} \\ \text{O} \text{ O} \end{array}$	49	0.91	73	0.95	73	0.95	IIA
Oxalic acid, diethyl ester	$\text{C}-\text{C} \begin{array}{c} \text{O} \\ \parallel \\ \text{O}-\text{C}-\text{O}-\text{C}-\text{O}-\text{C}-\text{C} \\ \text{O} \end{array}$	75	0.90	112	0.97	112	0.97	IIA
Diethyl sulphate	$\text{C}-\text{C}-\text{SO}_2-\text{C}-\text{C}$	104	1.01	135	1.11	135	1.11	IIA
14. Anhydrides								
Acetic anhydride	$\text{C} \begin{array}{c} \text{O} \\ \parallel \\ \text{O}-\text{C}-\text{O}-\text{C}-\text{C} \\ \text{O} \end{array}$	49	1.17	78	1.23	78	1.23	IIA
15. Amides								
N,N-Dimethyl formamide	$\text{C} \begin{array}{c} \text{H} \\   \\ \text{C}-\text{N}-\text{C}=\text{O} \\   \end{array}$	67	1.02	90	1.08	90	1.08	IIA

TABLE 1 (continued)

Name	Formula	Flash point (°C)	MESG (mm)	Temp (°C)	Corrected MESG (mm) (100 kPa, 20°C)	Group
16. <i>Cyclic ethers</i>						
Furan	$\text{O}-\text{C}=\text{C}-\text{C}=\text{C}$	-35	0.68	20	0.68	IIB
Tetrahydrofuran	$\text{O}-\text{C}-\text{C}-\text{C}-\text{C}$	-17	0.87	20	0.87	IIB
Tetrahydrofurfuryl	$\text{O}-\text{C}-\text{C}-\text{C}-\text{C}-\text{C}-\text{OH}$	75	0.80	100	0.85	IIB
2-Furfuraldehyde	$\text{O}-\text{C}=\text{C}-\text{C}=\text{C}-\text{C}-\text{H}$	59	0.88	94	0.94	IIA
1,3,5-Trioxalone	$\text{O}-\text{C}-\text{O}-\text{C}-\text{O}-\text{C}$	44	0.71	90	0.75	IIB
Morpholine	$\text{O}-\text{C}-\text{C}-\text{NH}-\text{C}-\text{C}$	38	0.92	58	0.95	IIA
Propane, 1-chloro-2,3-epoxy	$\text{O}-\text{C}-\text{C}-\text{C}-\text{Cl}$	32	0.95	58	1.00	IIA

application of these two temperature corrections to the MESG measurements at 100 kPa gives corrected room temperature and pressure MESG values differing, in most cases, by less than 0.02 mm, which is the experimental uncertainty of the apparatus. The tabulated room temperature and pressure MESGs have been obtained by applying the temperature correction derived from PTB's ethylene results, which gives a slightly lower value of the room temperature MESG, erring on the side of safety.

The correction is:

$$\text{Corrected MESG (100 kPa, 20}^\circ\text{C)} = \frac{\text{Corrected MESG (100 kPa)}}{1.1061 - (0.0071 \times T)}$$

where  $T$  is the experimental temperature in  $^\circ\text{C}$ .

## Results

The measurements are tabulated in Table 1. The flash point, the experimental temperature and MESG at that temperature, the MESG corrected to 100 kPa and  $20^\circ\text{C}$  and the grouping of the corrected MESG are given.

The groupings laid down the International Electrotechnical Commission [6] are as follows:

Group I	—	Firedamp only
Group IIA	—	MESG $\geq 0.9$ mm
Group IIB	—	$0.9 > \text{MESG} \geq 0.5$ mm
Group IIC	—	MESG $< 0.5$ mm

## Conclusions

An apparatus and procedure for the measurement of Maximum Experimental Safe Gaps have been described. The apparatus can be used to measure MESGs of gases and vapours up to temperatures of  $130^\circ\text{C}$ , widening considerably the number of compounds for which measurements can be obtained. The procedure has advantages in that it is both rapid and accurate. Measurements obtained are in close agreement with those from both the 8-litre apparatus and the 20-ml apparatus developed by PTB.

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