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**OFFSHORE TECHNOLOGY  
REPORT - OTO 97 810**

**ASSESSMENT OF THE ACCURACY  
OF THE DRAEGER TUBE METHOD  
FOR THE ESTIMATION OF MINERAL  
OIL IN BREATHING AIR SUPPLIED  
BY COMPRESSORS**

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## **FOREWORD**

This report is published by the Health and Safety Executive as part of a programme of work which was commissioned in support of the Offshore Safety Division's (OSD) diving research strategy. The full programme of work covers the period from the late 1970's to 1997; some reports from the programme have hitherto not been published.

Some research was sponsored by the Department of Energy prior to the transfer of their responsibilities for offshore safety to the Health and Safety Executive. Other studies were originally commissioned by OSD for internal use. It has now been decided to issue the reports relating to this work so that the information they contain is in the public domain.

In view of the extended period of the research programme, some reports may contain information or recommendations which have been superseded. The structure of others may not meet the standard now expected of an Offshore Technology series report. Nevertheless it is HSE's intention that all such documents should be in the public domain.

Assessment of the accuracy of the Draeger tube method for the estimation of mineral oil (as mist and vapour) in breathing air supplied by compressors.

Department of Energy project reference number TA93/22/25

1. Introduction.

Hydrocarbon oil mist and/or vapour is commonly measured in breathing air supplied by compressors using Draeger indicating tubes manufactured by Drägerwerk A G Lubeck FRG.

PCMU Harwell was contracted by the Department of Energy via MATSU (Mr D Robertson) to assess the accuracy of the Draeger tube measurements by comparison with other analytical techniques.

It was decided to make initial comparisons of Draeger tube measurements with infrared spectrophotometric determinations both carried out simultaneously on laboratory generated mists.

Experiments were mounted to devise suitable apparatus for the atomisation and containment of a compressor oil to yield relatively stable mists at concentrations up to 10 mg per M<sup>3</sup> as indicated by Draeger tubes. The mists were aspirated simultaneously through Draeger tubes and through tubes of similar dimensions containing a pre-washed plug of cellulose filter paper set in a position corresponding to the absorbing layer of the Draeger tubes.

The oil mist concentrations were estimated visually by two analysts in the case of the Draeger tubes by comparison with the published data and IR spectrophotometrically in solution from Freon 113 back-washing of the cellulose filters and comparison of aliphatic hydrocarbon absorption with prepared standards of the compressor oil in the same solvent.

2. Experimental Procedure

The oil mist/vapour generation and sampling apparatus is pictured diagrammatically in Figure 1 appended. Essentially the apparatus consisted of a domed Perspex glove box of approximately 130 litres capacity with an attached additional 20L volume Tedlar bag to act as a buffer during sampling. Oil aerosols were produced by atomising 'Castrol Perfect' compressor oil in the transfer port using nitrogen gas after preflushing with nitrogen. Atomisation was carried out for a period of 15 seconds to produce a barely visible cloud which was allowed to settle and equilibrate for periods of between 5 and 30 minutes. The Draeger tubes were prepared by snipping off end seals and clearing the sampling end to expose the full tube bore.

Cellulose filter-tubes were constructed of glass of the same bore as the Draeger tubes containing a plug made from a 7 cm diameter Whatman No.42 filter circle set to the same depth as the activated silica gel of the Draeger tubes. Papers were pre-washed with Halocarbon 113 using a Soxhlet extractor.

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Both Draeger Type 10/a-p 6728371 and cellulose filter tubes were inserted through small access ports cut in the side wall of the apparatus and sealed with silicone rubber. The sampling ends of the tubes were positioned within 3 cm of each other within the apparatus and at approximately the same depth. Mists and vapours were aspirated through the tubes at a constant rate of 1 litre per minute, measured by flowmeters, for 5 minutes to give sample volumes of 5 litres. (Note: The recommended maximum gas-flow rate quoted in the Draeger data sheet for 10/a-p tubes is 2 litres per minute when used with the Aerotest D 20700 sampling device. The mean flow-rate when using the 100 cm<sup>3</sup> bellows pump was measured at approximately 1 litre per minute for 10 strokes. Data sheet 10/a-p 6728371 is appended).

Draeger tube reactions were carried out and comparative measurements made by two analysts as described in the Data sheet.

The cellulose filter tube was removed from the mist chamber and the contents back-washed with successive aliquots of Halocarbon 113, (1,1,2 - trichlorotrifluoroethane), to 10 mls in graduated flasks. Infrared spectra of the solutions were recorded over the region 3100-2700 cm<sup>-1</sup> from a 5 mm pathlength cell fitted with calcium fluoride windows using a Perkin-Elmer model 683 spectrophotometer. Spectral information was stored on discs in the PE 3600 Data station and corrections were subsequently made for blank absorptions.

Standards of Castrol Perfect oil were prepared in the Halocarbon 113 and a calibration curve obtained of aliphatic -CH<sub>2</sub>- absorption of the oil at 2930 cm<sup>-1</sup> against concentration.

Blank determinations were undertaken on four cellulose packed tubes using the IR spectrophotometric method. 5 litre samples of laboratory air were also examined by both Draeger tube and IR methods.

Hydrocarbon vapour plus possible residual mist in the oil covered containment vessel was measured 60 hours after mist generation using a Draeger tube to establish an upper limit of vapour concentration. True vapours would probably not have been detected by the IR method.

### 3. Results

Comparative Draeger tube and IR results expressed as mg of mineral oil per M<sup>3</sup> are presented in Table 1 below. The infrared results were corrected for solvent background absorption but uncorrected for method blank (generally found to be less than 20 % of the total measured hydrocarbon content).

Interpretation of the Draeger tube results was somewhat subjective. Approximate, or ranges of values indicated, given in the table reflect the interpretation of at least two observers. Interpretations were in reasonable agreement.

Draeger tube measurements showed only low levels of hydrocarbon vapour (or possible trace residual mist) after an extended period of settlement of 60 hours.

Laboratory air blanks using both Draeger and IR methods produced similar low level figures.

Oil mist concentrations as measured by the infrared method were consistently higher than those obtained using Type 10/a-P Draeger tubes generally by a factor of between 3 and 7.

4. Conclusion and Discussion.

Comparison of results on oil mist concentration by the two methods indicated that the Draeger tubes gave significantly lower readings compared to the IR method. The reasons for the low results have not been fully investigated in this work although the following three possibilities come to mind:

(a) Incomplete chemical reaction of the reagents with the absorbed oil;

(b) Deposition of oil mist on the metal gauze and tube wall in front of the absorbing layer of silica gel;

(c) Incomplete absorption of the mist on the silica gel.

In view of the chemistry of the reaction, (given in the data sheet), (a) would seem unlikely.

During some preliminary experiments where the reagent inadvertently came into contact with the upper reaches of the tube, it was noticed that some dark coloration occurred which suggested the presence of oil on the tube wall. (b) above would, therefore, appear to be a contributory factor. Any such deposited oil in the case of the IR tubes would have been included in the analysis.

Pre-deposition of oil mists would depend on particle-size distribution and gas-flow velocity both of which were considered to be constant during sampling.

The effectiveness of trapping oil mists was not determined for either the Draeger tube or IR method.

Hydrocarbon vapours would not be expected to be efficiently absorbed by the cellulose filter of the IR tube. Total hydrocarbon as mist plus vapour would probably present slightly higher results than shown for mist in table 1 under the IR heading.

Information was sought from the UK Draegerwerk agents (Draeger Safety, Sunnyside Road, Chilsham, Bucks, HP5 2AR) concerning the method of calibration of their tubes but no useful information was forthcoming.

5. Further investigation work

As indicator tubes offer a convenient method for the assessment of oil mist/vapour, albeit suffering to some extent from the rather subjective interpretation of the colour intensity formed at the lower end of the concentration range, it may be worthwhile carrying out further comparative analyses to establish a more accurate Draeger tube calibration. Re-calibration should take into account particle size distribution, particularly with respect to respirable ranges, and sample flow rates. Replicate analysis would need to be undertaken

with an additional analytical method and account taken of trapping efficiencies.

Consideration could be given to the development of a simple alternative to the use of indicator tubes for the measurement of oil mist and vapour in breathing air from compressors.

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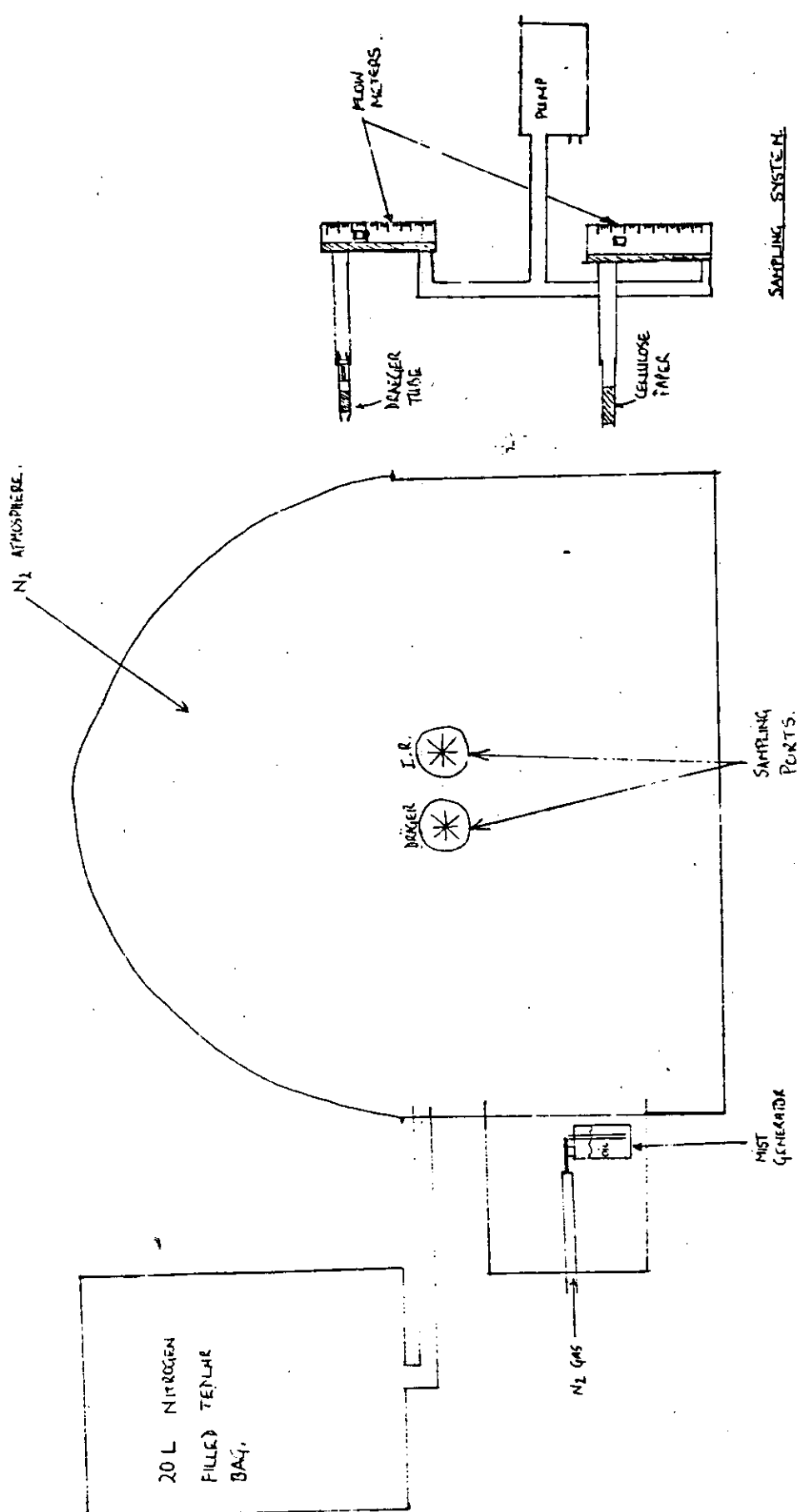


FIGURE ONE: MIST GENERATION AND SAMPLING APPARATUS.

TABLE I

## OIL MIST/VAPOUR CONCENTRATION COMPARISON OF DRAEGER TUBE AND IR RESULTS

SAMPLE	ATOMISATION TIME (SECS)	SETTLING TIME (MINS)	VOLUME SAMPLED (LITRES)	DRAEGER TUBE MEASUREMENT (mg/M <sup>3</sup> )	INFRARED DETERMINATION* (mg/M <sup>3</sup> )	RATIO IR: DRAEGER
BLANK CELLULOSE PACKED TUBE	-	-	-	-	1. 1.6 2. 5.6 3. 5.6 4. 2.8 Mean. 3.9	
LABORATORY AIR BLANK	-	-	5	<5	<3	
OIL COVERED CONTAINMENT VESSEL (Vapour + trace mist?)	-	60 HOURS	20	1. 1.3 2. 1.3	-	
MIST/VAPOUR 1.	15	5	5	APPROX. 10	33	3
MIST/VAPOUR 2.	15	5	5	5-10	32	6-3
" 3.	15	5	5	5-10	34	7-3
" 4.	15	10	5	<5	25	>5
" 5.	15	15	5	APPROX. 5	29	6
" 6.	15	30	5	<<5	12	>2

\* Corrected for solvent absorption

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**1 Allgemeines und Anwendungsbereich**

Dieses Röhrchen ist vorgesehen für die Messung des Ölgehaltes (Oldampf = Ölnebel) in Druckgasen.

Die Röhrchen sind mit der vorgeschriebenen Probenahmeeinrichtung zu verwenden (DRÄGER-Aerotest D 20700).

**Wichtiger Hinweis:**

Das mit diesem Röhrchen erhaltene Meßergebnis läßt nur Rückschlüsse auf den Ölgehalt der zur Messung entnommenen, d. h. entspannten Luftprobe zu. Zur Beurteilung des Ölgehaltes der gesamten Luft im Drucksystem sind zeitliche und räumliche Unterschiede aufgrund von Adsorptions- und Desorptionsvorgängen an Oberflächen zu beachten.

**2 Beschreibung**

Vgl. Abbildung 1

Bei der Analyse sollte der Volumendurchfluß der Luft durch das Röhrchen 2 Liter/min betragen.

- 1 und 2 zugeschmolzene Spitzen
- 3 Reagenzampulle (konz. Schwefelsäure)
- 4 Bruchstelle (doppelt gepunktet)
- 5 aufgeschumpfter Schlauch
- 6 Anzeigeschicht (weiß)
- 7 Pfeil (gibt die Stromungsrichtung der Prüfluft an)
- 8 Schreibfläche

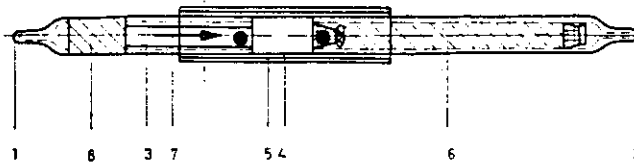


Fig. 1

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- 1 and 2 fused tips
- 3 reagent ampoule (conc. sulphuric acid)
- 4 breaking point (marked with two dots)
- 5 shrunk-on tubing
- 6 indicating layer (white)
- 7 arrow (indicates the direction of flow of the air sample)
- 8 writing surface

**3 Meßbereich (20°C, 1013 mbar)**

In absoluter Maßeinheit:

25 bis 100 µg (Mikrogramm) Luftverdichterschmieröl

Gemäß Farbstandard (Abb. 2) entspricht die Anzeige folgenden Farbstufen:

25 µg, 50 µg, 75 µg, 100 µg

(bezogen auf Luftverdichterschmieröl)

Legt man ein anzuwendendes Prüfluftvolumen im Bereich von 10 bis 250 Liter zugrunde, ergibt sich – auf die Konzentration bezogen – folgender Meßbereich:

Etwa 0,1 mg Öl/m<sup>3</sup> bis etwa 10 mg Öl/m<sup>3</sup>.

**4 Prüfung und Beurteilung des Ergebnisses**

- 4.1 Vorgeschriebenen, sauberen Druckminderer an Kompressor oder Druckluftflasche anschließen.
- 4.2 Druckminderer mit der zu untersuchenden Druckluft spülen (Dauer des Spülvorganges ggf. durch Vorversuche ermitteln).
- 4.3 Druckminderer öffnen und Volumenfluß auf 2 Liter/min einregulieren.
- 4.4 Spitzen des Drägerrohrchens öffnen.
- 4.5 Drägerrohrchen dicht in das Anschlußstück des Druckminderers einsetzen (Stoppur einschalten).
- 4.6 Nach Beendigung der Messung Prüfröhrchen aus Druckminderer herausnehmen und an der Bruchstelle (doppelt gepunktet) anknicken, so daß äußeres Glasrohr und innen eingebaute Reagenzampulle zerbrechen. (Vorsicht: Ampulleninhalt konzentrierte Schwefelsäure).
- 4.7 Ampullenflüssigkeit (konzentrierte Schwefelsäure) durch vorsichtiges Durchleiten der Prüfluft (ca. 10 Sekunden, 2 l/min) auf Anzeigeschicht verteilen.  
**Achtung:** Röhrchen nicht fest mit Druckminderer verbinden. Pfeil gibt die Stromungsrichtung an.
- 4.8 Auswertung: Verfärbung der Anzeigeschicht gemäß Farbstandard (Abb. 2) auswerten (Zwischenwerte schätzen).
- 4.9 Berechnung des Meßergebnisses

$$\frac{\text{ermittelte Farbstufe in Mikrogramm}}{\text{Prüfluftmenge in Liter}} = \text{mg Öl/m}^3$$

**4.10 Hinweis**

Liegt die Farbintensität der Anzeige außerhalb des Bereiches von 25 bis 100 µg, so ist der gesamte Test – beginnend mit 4.3 – erforderlichenfalls zu wiederholen, dazu ein anderes Luftvolumen durch die Röhrchen leiten.

Beispiele:

zu erwartende Ölkonzentration in mg/m <sup>3</sup>	Durchgeleitetes Volumen in Liter	Anzeige gemäß Farbstufe
0,2	125	etwa 25
0,3	80	etwa 25
0,5	50	etwa 25
1	25	etwa 25
2	50	etwa 100

**5 Bemerkungen**

Auch nach negativem Ergebnis kann das DRÄGER-Röhrchen nicht wieder verwendet werden. Die Anzeige ist mehrere Stunden haltbar, wenn die Röhrchen mit Gummikappen verschlossen werden.

**6 Einfluß der Umgebungsbedingungen auf das Meßergebnis**

- 6.1 Temperatur: Die DRÄGER-Röhrchen können in einem Temperaturbereich von 10 bis 30°C verwendet werden.
- 6.2 Feuchtigkeit: Feuchtigkeit verdünnt das Reagenzsystem. Enthält die untersuchte Luft mehr als 1000 mg Wasser/m<sup>3</sup>, kann dieser Verdünnungseffekt zu einer nicht mehr eindeutigen Anzeige führen, daher vor der Dümung immer eine Feuchtigkeitsbestimmung mit Wasserdampf-Prüfröhrchen vornehmen.

**7 Reaktionsprinzip**

Oldampf - Ölnebel werden an aktiviertem Silicagel adsorbiert. Nach Abschluß der Sorption wird das Öl durch konzentrierte Schwefelsäure in Gegenwart eines Katalysators zersetzt. Dabei entstehen dunkelgelarbte Reaktionsprodukte, deren Farbintensität gemessen wird.

**8 Ergänzende Information:**

Das Öl-Röhrchen ist auch zur Untersuchung der Luft in Arbeitsräumen in Verbindung mit der DRÄGER-Balgpumpe geeignet. Messungen in Raumluft sind mit max. 100 Huben der Pumpe auszuführen; bis zu diesen Hubzahlen beeinflusst die Luftfeuchtigkeit das Meßergebnis nicht.

**9 Vorgesehene Verbrauchszelt**

Verbrauchsdatum und Lagertemperatur (vgl. die Angaben auf der Banderole).

Verbrauchte DRÄGER-Röhrchen nicht achtlos fortwerfen, damit sie nicht in Kinderhände gelangen! Inhalt atzt!

**1 General and Application**

This tube is intended for measurement of the oil content (oil vapour and oil mist) in compressed gases.

The tubes are to be used in conjunction with the prescribed sampling device (DRÄGER-Aerotest D 20700).

**Important information:**

The result of measurement obtained with this tube can be used only for determination of the oil content of the sample used for measurement, i. e. air relieved of pressure. For evaluation of the oil content of all the air in the pressurised system, local difference and differences in time must be noted, because of adsorption and desorption processes on surface.

**2 Description**

See illustration 1

During analysis, the rate of flow of air through the tube should be 2 litres/min

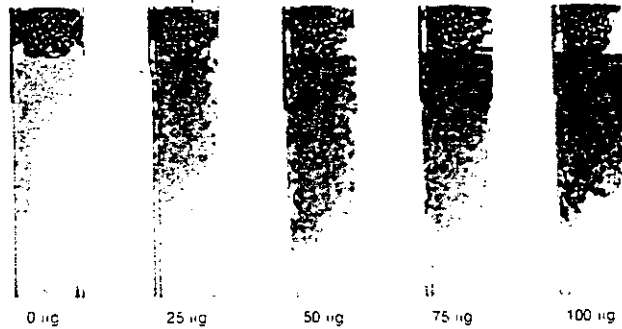


Fig. 2

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**3 Range of Measurement (20°C, 1013 mbar)**

In absolute units:

25 to 100 µg (micrograms) air compressor lubricating oil.

In accordance with the colour standard (Fig. 2), the indication corresponds to the following colour stages:

25 µg, 50 µg, 75 µg, 100 µg

(relative to air compressor lubricating oil)

If we take as a basis a test volume between 10 and 250 litres, the following range of measurement – related to concentration – results:

app. 0,1 mg oil/m<sup>3</sup> to app. 10 mg oil/m<sup>3</sup>

**4 Test and Evaluation of the Result**

- 4.1 Connect the prescribed, clean pressure reducer to the compressor or compressed-air cylinder.
- 4.2 Flush the pressure reducer with the air to be investigated (if necessary, determine the preflushing time experimentally in advance).
- 4.3 Open pressure reducer and adjust volume flow to 2 litre/min.
- 4.4 Open tips of DRÄGER Tube
- 4.5 Insert DRÄGER Tube tightly into the connecting piece of the pressure reducer (set stopwatch).
- 4.6 After completing measurement, remove the detector tube from the pressure reducer and bend it at the breaking point (marked with two dots), so that the outer glass tube and the inner reagent ampoule break. (Take care, the ampoule contains concentrated sulphuric acid).
- 4.7 Force ampoule liquid (concentrated sulphuric acid) onto the indicating layer by carefully leading the air sample through it (about 10 seconds, 2 l/min).  
**Note:** Do not connect tube firmly to pressure reducer. Arrow indicates direction of flow!
- 4.8 Evaluation: Evaluate discoloration of the indicating layer in accordance with colour standard (Fig. 2). Estimate intermediate values.
- 4.9 Calculation of the result of measurement

$$\frac{\text{colour stage determined in micrograms}}{\text{air sample volume in litres}} = \text{mg oil/m}^3$$

**4.10 Note**

If the intensity of colour of the indication lies outside the range of 25 to 100 µg, the entire test – starting at 4.3 – must be repeated if necessary, leading a different air volume through the tube.

Examples:

Oil concentration to be expected in mg/m <sup>3</sup>	Volume led through, in litres	Indication acc. colour stage
0,2	125	app. 25
0,3	80	app. 25
0,5	50	app. 25
1	25	app. 25
2	50	app. 100

**5 Remarks**

The DRÄGER Tube cannot be used again, even after a negative result. The indication lasts for several hours if the tubes are sealed with rubber caps.

**6 Influence of Ambient Conditions on the Result of Measurement**

- 6.1 Temperature: The DRÄGER Tubes can be used in a temperature range of from 10 to 30°C.
- 6.2 Humidity: Moisture dilutes the reagent system. If the air sample contains more than 1,000 mg water/m<sup>3</sup>, this diluent effect can lead to an oil indication which is no longer clear. Consequently, determination of the moisture content with a water vapour detector tube must always be carried out before the oil measurement.

**7 Reaction Principle**

Oil vapour - oil mist are adsorbed on activated silica gel. After completion of the adsorption, the oil is decomposed by concentrated sulphuric acid in the presence of a catalyst. This leads to the formation of dark-coloured reaction products, the colour intensity of which is measured.

**8 Additional Information:**

In connection with the DRÄGER-bellows pump the Oil Tube can also be used for investigation of air in workrooms. Measurement of room air is performed with a maximum of 100 pump strokes, up to this number of pump strokes humidity has no influence on the result of measurement.

**9 Shelf Life**

For expiry date and storage temperature see data on label of the box.

Do not carelessly discard used Dräger Tubes, since they must not fall into the hands of children.

The contents are corrosive.