



## MDHS 32

Methods for the  
Determination of  
Hazardous Substances

December 1987

# Diocetyl phthalates in air

Laboratory method using Tenax sorbent tubes, solvent desorption and gas chromatography

## INTRODUCTION

### Properties and uses

1 Di(2-ethylhexyl)phthalate,  $C_{24}H_{38}O_4$ , synonyms *bis* (2-ethylhexyl) phthalate or *diocetyl phthalate* in common usage, is a light-coloured odourless, combustible liquid of molecular weight 390 and density 0.99g/ml. Its boiling point is 231°C at 5mm Hg (6.7 kPa) and it has a very low vapour pressure at ordinary temperatures. It is insoluble in water, but miscible with cyclohexane. It is used widely as a plasticiser in various types of synthetic plastics and resins.

2 Another large-use plasticiser is 'diisooctyl' phthalate, made from alcohols derived from propylene-butadiene codimer or propylene trimer by means of the oxo reaction. ('iso' in plasticisers, refers to highly-branched products, often, as in this case, a mixture of isomers). Its physical properties are similar to those of di(2-ethylhexyl) phthalate.

### Toxicity

3 Di(2-ethylhexyl) phthalate and similar phthalates are substances of relatively low acute and subacute toxicity. Mutagenicity tests have proven negative in a variety of systems. There is some evidence of carcinogenicity (liver tumours) in both rats and mice, but by the oral route only. There is no direct evidence of human carcinogenicity.

### First aid

4 Following massive exposure to hot fume, the patient should be removed to a clean atmosphere and artificial respiration given if breathing has ceased. Contaminated clothing should be removed. The patient should be transferred to hospital.

### Analytical methods

5 This is not a reference method in the strict analytical sense of the word. A number of analytical methods are available for the determination of diocetyl phthalates in air, including two methods recommended by the analytical working group on phthalate and adipate plasticisers.<sup>1,2</sup> One of these methods is similar to that presented here; the other uses a silica gel sorbent tube and UV spectrophotometric analysis. With the exception of a few cases where an exposure limit is linked to a specific method (eg for rubber fume or asbestos), the use of methods not included in the MDHS series is acceptable provided they have the accuracy and reliability appropriate to the application.

## SCOPE

6 The method is described for the determination of the time-weighted-average concentrations of di(2-ethylhexyl) phthalate and diisooctyl phthalate in workplace atmospheres. The method is suitable for sampling over a period of 8 h, but may be used for sampling over shorter periods by increasing the sampling flow rate to a maximum of 500ml/min. The method does not distinguish between di(2-ethylhexyl) phthalate and diisooctyl phthalate because of their closely corresponding retention times. Thus if they are both present, the method gives a total value. The method is suitable for the determination of these compounds present in air as vapour or aerosol, but does not distinguish between the two forms. The aerosol fraction collected will not necessarily be the 'inspirable fraction'. Although described for the determination of personal exposure, the method may be used for fixed location monitoring by suitable modification.

7 Although described for a Tenax tube made of metal, smaller 'NIOSH'-type tubes made of glass and containing two sections of Tenax (30 mg and 15 mg) are equally suitable (see para 20).

8 The method is suitable for the measurement of airborne diocetyl phthalates in a concentration range of approximately 0.5 to 25mg/m<sup>3</sup> for samples of 50 litres of air. The method may be applicable to other, similar, phthalates and also to some other plasticisers, eg adipates.

9 The upper limit of the useful range is set by the sorptive capacity of the Tenax sorbent. However, this capacity is very large, and in practice, saturation of the sorbent is unlikely to occur.

10 The lower limit of the useful range depends on a number of constraints, including the noise level on the detector (para 23), blank levels of phthalates in the Tenax tube or cyclohexane, contamination by phthalates of the apparatus used (para 42), adequate desorption efficiency (para 39) and interference of the solvent peak in the gas chromatographic analysis.

11 The method is similar to that described in Ref 1. It has been evaluated for both the tubes described in para 19 and the smaller glass tubes described in para 20 in sampling diocetyl phthalate from a laminating test rig.<sup>3</sup> Sampling efficiency for both types of tube was approximately 82%, and combined sampling and analytical error approximately 18%.

12 The bias of the method is expected to be better than 5%, if due correction is made for sampling efficiency (paras 11 and 32).

## Interferences

13 Any compound that co-elutes with the analytes of interest at the operating conditions chosen by the analyst is a potential interferent; alteration of conditions may remove this interference. Correspondence of retention time on a single column cannot be regarded as proof of identity. Details of likely interferents are given in the Appendix.

14 When interfering compounds are known to be present in the air, or are suspected of being present, the identity or suspected identity of the compounds should be transmitted with the sample.

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## PRINCIPLE

15 A measured volume of sample air is drawn through a glass or metal tube packed with Tenax porous polymer sorbent. The dioctyl phthalates are adsorbed on the Tenax and thus are removed from the flowing air stream. The collected phthalates are desorbed by cyclohexane and the solution is analysed with a gas chromatograph equipped with a flame ionization detector.

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## REAGENTS

16 During the analysis, use only reagents of recognised analytical reagent grade.

### Dioctyl phthalates

### Cyclohexane

17 The cyclohexane (C<sub>6</sub>H<sub>12</sub>) should be of chromatographic quality. It must be free from compounds co-eluting with the analyte of interest.

### Tenax

18 Tenax GC porous polymer sorbent (Tenax is a registered trade mark of Akzo Research Laboratories), particle size 0.18-0.25mm (60-80 mesh) should be used. The sorbent should be preconditioned by heating in an inert atmosphere at 300°C.

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## APPARATUS

### Tenax tubes

19 Typically, adsorbent tubes are constructed of stainless steel tubing, 6mm (¼ inch) OD, 5mm ID and 90mm long. One end is marked, for example by a score ring 10mm from the end. The tubes are packed with preconditioned Tenax to within about 10mm of each end. Tubes contain typically about 200mg Tenax. The polymer is retained by silanized glass wool plugs. Empty tubes are available commercially, but must be packed by the user.

20 Alternatively, 'NIOSH'-type tubes may be used. These consist of glass tubes with both ends flame-sealed; 70mm long with 6mm OD and 4mm ID, containing two sections of preconditioned Tenax separated by a 2mm section of urethane foam. The first section contains 30mg Tenax and the second section, 15mg; for this application both sections

are desorbed together as for the single-section metal tube (para 34) and a complete second tube used for a back-up, if desired (para 32).

### End caps

21 Metal sorbent tubes should be provided with metal end-caps with PTFE or Viton seals. Glass tubes should be provided with polythene end caps. Materials used for sealing the tubes must be free from phthalate plasticisers.

### Pump

22 A pump is required which is capable of being worn by a person while carrying out his normal work, and capable of running continuously for 8h at the recommended flow rate (para 30). The total volume of air sampled by the pump over the recommended sampling period should be with ± 5% of the calculated volume. The pump should be certified intrinsically safe if it is to be used in areas where a flammable gas hazard may be present.

### Gas chromatograph

23 A gas chromatograph designed for packed column operation with on-column injection and fitted with a flame ionization detector is suitable.

24 A variety of chromatographic columns may be used for the analysis of dioctyl phthalate in solution. The choice will depend largely on which compounds, if any, are present, that might interfere in the chromatographic analysis. A suitable choice might be a 1m by 2mm ID glass column packed with 5% silicone (eg OV101) on silanized, acid-washed Chromosorb W or 2m by 2mm ID glass column packed with 3% Dexsil 300 on Chromosorb G. Both columns are operated at 260°C. Di(2-ethylhexyl) phthalate elutes from the latter column in about 3min at a carrier flow rate of 30ml/min.

### Glassware

25 Graduated glass septum vials, 3ml capacity and with internal cone-shaped bottom sections and PTFE-lined septa are suitable.

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## PROCEDURE

### Calibration of sampling pumps

26 Measurement of the volume of air sampled may be a significant source of error in the final calculation of vapour-in-air concentrations. Calibrate the sampling pump with a representative Tenax tube in line, using an appropriate external calibrated meter. Note that at high flow rates, tubes may have a significant back-pressure.

### Tenax tubes

27 Prior to use, the metal sorbent tubes (para 1) should be conditioned by heating under inert carrier gas to 300°C and maintaining that temperature for 10 min. Representative tubes should then be analysed (paras 33 to 38) to ensure that the blank is adequate (para 42). If the blank is inadequate, tubes should be reconditioned. Once a sample has been analysed, the tube can be reused after first removing the bulk of the cyclohexane by flushing with inert

carrier gas at room temperature, passing about 2 litres of carrier gas at 100ml/min, and then reconditioning at 300°C for 10 min. Glass tubes (para 20) are preconditioned, but are not conveniently reusable. The phthalate blank of a representative tube should be checked and the batch rejected if the blank is inadequate.

### Collection of samples

28 Immediately before sampling, remove the end caps from the sorbent tube and connect it to the sampling pump with appropriate tubing. The tube should be oriented such that the end of the tube opposite to the marked end is connected to the pump. The tube should be positioned vertically with air passing downwards to minimise channelling. Ensure that all connections are free from leaks.

29 When used for personal sampling, the tube should be mounted in the worker's breathing zone, for example on his lapel. The pump is attached to the worker as appropriate to minimise inconvenience. The sampled air should not pass through any tubing prior to the sampling tube.

30 Draw a measured volume of air through the sorbent tube, and immediately cap the tube with appropriate end caps (para 21). The sample volume should be a minimum of 50 litres. This volume is equivalent to an air flow rate of approximately 100ml/min for an 8h sample. For sampling over shorter periods, the flow rate may be increased in proportion, but should not exceed 500ml/min. It is advisable to store used tubes in airtight sealed containers to prevent additional contamination.

31 Sample blanks should be prepared by using tubes identical to those used for sampling and subjecting them to the same handling procedure as the sample tubes except for the actual period of sampling.

### Breakthrough volume

32 Although the saturation capacity for Tenax for dioctylphthalate is very large, in practice, a small amount of breakthrough tends to occur. In a field test<sup>3</sup> conducted over a period of 6 to 7h, approximately 18% of the total phthalate was found on a second tube in series with the first, i.e. the sampling efficiency of the first tube was 82%. Breakthrough was unaffected statistically by type of tube, flow rate, (between 20 and 100 ml/min) or concentration (between 0.15 and 85 mg/m<sup>3</sup>). It is recommended that a figure of 82% for sampling efficiency is assumed, or a second, back-up, tube used in the test.

### Desorption

33 Desorption should be carried out in a clean atmosphere in a fume hood. Remove the end caps from the sample tube and place the tube in a vertical position above a graduated septum vial. The tube should be placed such that the subsequent elution will take place in an opposite direction to that of sampling, i.e. the marked end should be at the bottom.

34 Place 0.5 ml of cyclohexane in the open upper end of the tube and allow the solvent to percolate through, collecting the eluate in the sample vial. Repeat with two further portions of 0.5 ml cyclohexane, but do not collect more than a total of 1.0 ml of eluate. If necessary, make the volume of eluate collected up to 1.0 ml with additional cyclohexane. Stopper, and agitate the vial to mix. Desorb the sample blanks and the samples used to determine desorption efficiency (para 39) in the same way.

### Preparation of standard solutions

35 Standard solutions of the dioctyl phthalates in cyclohexane should be prepared by serial dilution. The concentration range of interest should exceed the concentration range of desorbed samples (paras 33 to 34). Prepare fresh standard solutions with each batch of samples.

36 It may be necessary to prepare a few standards covering a wide range of concentrations (say 10 µg/ml to 1 mg/ml) in order to determine the approximate concentration range of the desorbed samples and hence the concentration range of interest. Further standards can then be prepared if necessary.

### Analysis

37 Inject into the gas chromatograph a known fixed volume of each standard solution (para 35) in the range 1 to 5 µl. A standardised injection technique should be used such that reproducible peak heights or areas are obtained. Prepare a calibration graph of peak response against analyte concentration. The calibration graph may be non-linear.

38 Inject into the gas chromatograph the same fixed volume of the sample solution. Determine the peak response and read from the calibration graph the concentration of the analyte in the desorbed sample. Analyse the sample blank and the samples used to determine desorption efficiency (para 39) in the same way.

### Determination of desorption efficiency

39 The desorption efficiencies of dioctyl phthalates from Tenax can vary with the type and batch of Tenax used. Thus it is necessary to determine for each batch of tubes the desorption efficiency over the sample concentration range. For many compounds, this would normally be done by sampling from a standard vapour atmosphere and determining the recovery, but standard atmospheres of phthalate plasticisers are extremely difficult to maintain. The following procedure should thus be used.

40 Prepare a standard solution of dioctyl phthalate in cyclohexane at a concentration of 50 mg/ml. Use a microlitre syringe to inject a range of aliquots of this solution into a series of tubes, whilst maintaining a purge of inert gas through the tube at approximately 100 ml/min. It is convenient to do this with the aid of a gas chromatographic injection adapter (Fig 1). The injection and flow of gas should be in the same direction as for sampling, i.e. inject into the marked end of the tubes. A typical range of aliquots is 1, 2.5 and 10 µl, but this range should exceed the concentration range of the desorbed samples. Three samples at each tube load level are sufficient. After each injection, maintain the purge for 2 to 5 min, then remove and cap the sampling tube.

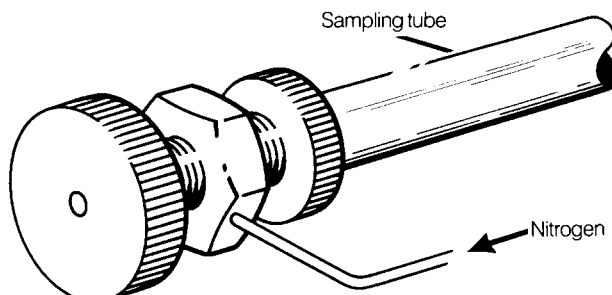


Fig 1 Injection adapter for preparation of standards for the determination of desorption efficiency

41 The desorption efficiency equals the weight (in  $\mu\text{g}$ ) recovered from the tube divided by the weight (in  $\mu\text{g}$ ) applied. Plot the desorption efficiency values against the weight recovered for each tube load level (DE curve). If the desorption efficiency at the sample load level is less than 0.75 (75%) the result should be discarded.

### Blanks

42 Phthalate plasticisers are common contaminants of many laboratory materials and special care must be taken to check the quality of materials to avoid sample contamination. An examination of the sample blanks will indicate the level of any contamination. This blank level is acceptable if it is no greater than the equivalent of 2.5  $\mu\text{g}$  of dioctyl phthalate per tube.

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## CALCULATIONS

43 Calculate the weight, in  $\mu\text{g}$ , of each analyte in the sample by comparison with standard solutions. Correct for blanks, sampling efficiency and desorption efficiency as follows:

$$\begin{array}{l} \text{Concentration of} \\ \text{analyte in air} \\ \text{(mg/m}^3\text{)} \end{array} = \frac{(m - m_{\text{blank}})}{\text{SE} \times \text{DE} \times V}$$

where

$m$  = weight ( $\mu\text{g}$ ) of phthalate on sample tube

$m_{\text{blank}}$  = weight ( $\mu\text{g}$ ) of phthalate on blank

SE = sampling efficiency (para 32)

DE = desorption efficiency, as read from the DE curve, taking  $m$  as the weight recovered

$V$  = volume of air sampled (litres)

44 Strictly speaking, separate desorption efficiencies should be made for  $m$  and  $m_{\text{blank}}$ , but in practice, the errors involved in not doing so are very small.

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## REPORT

45 Report the analyte-in-air concentration(s) to the nearest  $\text{mg/m}^3$ .

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## ADVICE

Advice on this method and the equipment used can be obtained from the Health and Safety Executive, Occupational Medicine and Hygiene Laboratory, 403-405 Edgware Road, London NW2 6LN (telephone 01-450 8911).

The Health and Safety Executive wishes, wherever possible, to improve the methods described in this series. Any comments that might lead to improvements would therefore be welcome and should be sent to the above address.

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## REFERENCES

- 1 British Plastics Federation/Chemical Industries Association. Time-weighted-average measurements of airborne dioctyl phthalates by gas chromatography. Analytical Working Group on Phthalate and Adipate Plasticisers, draft method 1. (1981).
- 2 *ibid.* TWA measurements of airborne dioctyl phthalates by UV spectrophotometry. Analytical Working Group on Phthalate and Adipate Plasticisers, draft method 2. (1981).
- 3 National Institute for Occupational Safety and Health. *Documentation of the NIOSH Validation Tests*. DHEW (NIOSH) Publication 77-185 (1977).

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## OTHER USEFUL REFERENCES

Kirk-Othmer. *Encyclopedia of Chemical Technology*. Eds. H.F. Mark et al, second edition (1970). Vol 15, p 456 (part of article on phthalic acid) Wiley-Interscience: New York.

National Institute for Occupational Safety and Health/Occupational Safety and Health Administration. *NIOSH/OSHA Occupational Health Guidelines for Chemical Hazards*. DHHS (NIOSH) Publication 81-123 (1981).

Patty, F.A. *Industrial Hygiene and Toxicology*. Eds. D.W. Fassett and D.D. Irish, third edition. 3 Vols. Interscience Publishers: New York, 1978-82.

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## APPENDIX

### Interferences

The procedure described in the method has been validated for the determination of dioctylphthalates. In recognition however, of the fact that the method may also be applicable to other phthalates, some other plasticisers (eg adipates), and may be used under conditions when mixtures may be present, 'interference' data has been compiled.

The following table summarises data obtained by subjecting 1-2  $\mu\text{l}$  quantities of a range of dilute plasticiser solutions in cyclohexane to the chromatographic procedure using a 2m 3% Dexil column at 260°C. Corrected relative retention values have been calculated to facilitate comparisons. The figure in brackets represents the approximate percentage of each constituent in each particular plasticiser.

As an example the table clearly indicates that di 2-ethyl-hexylphthalate and di-3,5,5' trimethylhexylphthalate are resolved and can be determined in the presence of each other. Dialphanol adipate, however, is shown to interfere in the determination of di iso-octylphthalate and it would thus be better to use the UV method or high performance liquid chromatography for the analysis.

CORRECTED RETENTION TIMES RELATIVE TO DI (2-ETHYLHEXYL) PHTHALATE 2.00m (1/4" OD GLASS) 3% DEXSIL COLUMN TEMPERATURE 260°C

CORRECTED RELATIVE PLASTICISER	400 to 499	500 to 599	600 to 699	700 to 799	800 to 899	900 to 999	1.00 to 1.09	1.10 to 1.19	1.20 to 1.29	1.30 to 1.39	1.40 to 1.49	1.50 to 1.59	1.60 to 1.69	1.70 to 1.79	1.80 to 1.89	1.90 to 1.99	2.00 to 2.49	2.50 to 2.99	3.00 to 3.49	3.50 to 3.99	4.00 to 4.99	5.00 to 5.99	6.00 to 6.99	Greater than 7.00						
DIOCTYL (2-Ethylhexyl)							1.00 (99+)	(3.07 min)																						
DI n-HEPTYL							1.03 (97+)																							
DI n-DECYL																								5.17 (99+)						
DI sec-OCTYL						985 (99+)																								
DINONYL (3,5,5-Trimethylhexyl)										1.38 (90)			1.69 (10)																	
DIPHENYL									1.23 (99+)																					
DI p-TOLYL																		2.27 (99+)												
DIBENZYL																		2.12 (99+)												
BUTYL BENZYL			666 (95)																											
DICYCLOHEXYL			666 (13)					1.13 (87)																						
DIOCTYL TETRAHYDRO (2-Ethylhexyl)					820 (96+)																									
DIBUTYL GLYCOL (Butoxyethyl)	413 (25)				823 (73)																									
DICYCLOISOCTYL							1.04 (28)	1.13 (24)	1.26 (48)																					
DIISOCTYL			692 (1.5)			983 (24)		1.15 (47)		1.39 (27.5)																				
DIISONONYL ('HEXAPLAS' DIMP)							1.04 (3.5)		1.22 (11.5)	1.37 (74)			1.61 (9.5)																	
DIISODECYL								1.16 (5)		1.39 (5)																				
DIALPHANOL (Alkyl)			677 (10)		843 (28)		1.09 (43)			1.39 (16)			1.79 (3)																	
'LANKROFLEX' OPN (AGS Esters/DIOP)	419 486 568					983		1.16		1.39																				
'PLIABRAC' 610 (Mixed linear C <sub>8</sub> -C <sub>10</sub> alcohols)			600 (5)				1.03 (17)						1.77 (33)					3.01 (30)					5.16 (15)							
'PLIABRAC' 810 (Mixed linear C <sub>8</sub> -C <sub>10</sub> alcohols)							1.03 (8)	1.15 (2.5)		1.37 (4)			1.76 (48)						3.01 (29)				5.17 (4)							
'LANKROFLEX' 79 LP (Mixed linear C <sub>7</sub> -C <sub>9</sub> alcohols)					836 (5)		1.03 (30)			1.35 (36)			1.76 (22)				2.31 (6)		3.01 (1)											
'BISOFLEX' 1911 P (Mixed linear C <sub>8</sub> -C <sub>11</sub> alcohols)																	2.34 (2)		3.07 (9)	3.96 (24)		5.17 (34)	6.74 (22)	8.82 (6)						
'SANTICIZER' 711 (Mixed C <sub>7</sub> , C <sub>8</sub> , C <sub>9</sub> alcohols)					839 (5.5)		1.03 (10)	1.17 (2)			1.42 (10)			1.77 (17)			2.42 (10.5)		3.05 (22)		4.11 (5)	5.18 (12)	6.92 (1)	8.84 (4)						
'SANTICIZER' 261 (Octyl benzyl)			664 (2)					1.19 (12)			1.46 (36)		1.69 (2)		1.84 (3)	1.99 (11)		2.52 (34)												
TRIPHENYL				770 (98)		908 (2)																								
TRICRESYL									1.29 (3)			1.53 (15)	1.66 (30)			1.84 (29)		2.08 2.28 2.54 (15) (6) (2)												
TRIXYLENYL																1.87 (2.5)		2.06 2.26 2.48 2.75 3.40 (5) (13) (17) (52) (10)												
'REOFOS' 50 (Ternary)				776 (25)			1.06 (40)				1.42 (7)	1.58 (6)		1.73 (10)				2.15 (10)												
'REOFOS' 65				777 (12)			1.06 (45)				1.42 (11)	1.58 (3.5)		1.72 (9.5)				2.15 (15)	2.79 (2)	3.44 (1)										
'REOFOS' 90				781 (5)			1.06 (33)				1.42 (24)	1.58 (2)		1.73 (6)				2.16 (21)	2.80 (8)	3.44 (2)		4.39 (2)								
'SANTICIZER' 141 (Octyldiphenyl)				778 (99+)																										
DIOCTYL SEBACATE (2-Ethylhexyl)						994 (99+)																								
DINONYL SEBACATE (3,5,5-Trimethylhexyl)																	1.94 (2)		2.57 (96+)											
DIBENZYL SEBACATE			613 (1)																											
DIOCTYL ADIPATE (2-Ethylhexyl)			616 (99+)																											
DINONYL ADIPATE (3,5,5-Trimethylhexyl)					806 (99+)																									
DIALPHANOL ADIPATE (Alkyl)	462 (11)	545 (15)	608 (19)	719 (19)	809 (21)	953 (11)			1.27 (2)																					
DIISOCTYL ADIPATE		548 (13)	656 (50)	758 (36)																										
DIISOCTYL AZELATE									1.21 (2)	1.35 (12)		1.57 (44)			1.83 (37)															
'BISOFLEX' 104 (Mixed succinates)		524 (29)				917 (33)																								
'CITROFLEX' AB (Acetyl tri(2-ethylhexyl) citrate)		524 (3)				928 (3)																								
MESAMOL	428 (5)	.506 (2)	578 (5)	682 (2)	740 (5)	966 (22)			1.26 (25)	1.39 (8)			1.61 (10)		1.84 (10)		2.15 (2)	2.34 (4)												

PHTHALATES

PHOSPHATES

MISCELLANEOUS

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