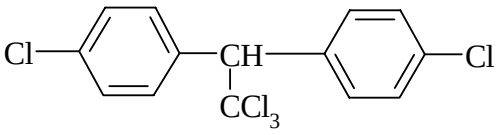
p,p'-DDT
3



ISO common name	p,p'-DDT
Chemical name	1,1,1-Trichloro-2,2-bis(4-chlorophenyl) ethane (IUPAC, CA; *50-29-3*)
Empirical formula	$C_{14}H_9Cl_5$
RMM	354.5
m.p.	108.5 °C
v.p.	1.7×10^{-5} Pa at 20 °C
d^{20}	1.54
Solubility	In water 1.2×10^{-3} mg/l; in acetone 500 g/l; diethylether 270 g/l; ethanol 60 g/l; benzene 770 g/l; chloroform 31 g/l; cyclohexanone 1 kg/l; dioxane 1 kg/l; methanol 40 g/l; dichloromethane 850 g/l; trichloroethene 720 g/l; xylene 600 g/l (all at 27 °C)
Description	Colourless crystals. The technical product is a waxy solid, containing about 30% *o,p'*-DDT
Stability	Loses hydrogen chloride in solutions of alkalis and organic bases. Iron aluminium and UV light promote the decomposition
Formulations	Wettable powders, emulsifiable concentrates and dustable powders

p,p'-DDT TECHNICAL
*3/TC/M/-

1 Sampling. Take at least 100 g.

2 Identity test. Use the GLC method below. The relative retention time of p,p'-DDT with respect to the internal standard for the sample solution should not deviate by more than 1% from that for the calibration solution.

3 p,p'-DDT

OUTLINE OF METHOD The sample is dissolved in chloroform with 2,2'-dinitrobiphenyl as internal standard added. The p,p'-DDT content is determined by gas chromatography with flame ionization detection.

REAGENTS

Chloroform

2,2'Dinitrobiphenyl internal standard

Internal standard solution. Weigh about 2.8 g 2,2'-dinitrobiphenyl into a volumetric flask (100 ml), dissolve in, and dilute to volume with chloroform. Mix well.

p,p'-DDT standard of known purity. Purity at least 995 g/kg.

Calibration solutions. Weigh (to the nearest 0.1 mg) about 150, 200 and 300 mg quantities (s mg) of the p,p'-DDT standard directly into separate stoppered conical flasks (50 ml) equipped with teflon-lined screw caps. To each flask add by pipette 5 ml of internal standard solution and 20 ml of chloroform. Cap each flask tightly and gently swirl the contents of each flask for 1 minute using a rotational motion of the wrist. Allow each flask to stand at least 5 minutes and then shake for 1 minute, either by hand or using a reciprocal shaker. Ensure that no leaking occurs around the cap. Allow each flask to stand for 30 minutes. Label the three calibration solutions "A", "B", and "C", respectively. Solution B is the working calibration solution for gas chromatography; solutions A and C are used to check the linearity of the gas chromatograph. These solutions are stable for 4 weeks if kept tightly sealed and under refrigeration. Allow the solutions to warm to room temperature before use. The supply of solution B can be replenished from time to time without preparing new supplies of solutions A and C, provided the linearity specifications described in section *(b)* can be met.

* CIPAC-WHO method 1990. Prepared by Mr F.C. Churchill, enters for Disease Control, Atlanta, USA.

APPARATUS

Gas chromatography suitable for on-column injection and equipped with a flame ionization detector
Column 1.83 m × 2 mm (i.d.) glass, packed with 5% OV-210 on Chromosorb W-HP, 100-200 mesh

PROCEDURE

(a) Operating conditions (typical):

Column temperature	170 °C
Injection port temperature	240 °C
Detector temperature	250 °C
Flow rate carrier gas	nitrogen, 30 ml/min.
Injection volume	2 µl
Number of theoretical plates	at least 2500
Retention times	p,p'-DDT : 13.5 minutes internal standard : 21 minutes o,p'-DDT : 8.5 minutes

(b) Linearity check. Check the gas-liquid chromatograph for linearity at least once a week, and whenever new calibration solutions are prepared or a column, new or used, is installed in the instrument.
Using digital integration for peak area measurements, determine the appropriate attenuation setting and the quantity (between 2 and 4 µl) of calibration solution B that must be injected to yield an area count of at least 100,000 (optimum electrometer output with acceptable noise level). The attenuation setting quantity so determined should be used for all samples and calibration solutions in the set.
Inject triplicate aliquots of appropriate volume (as determined above) of calibration solution A, B and C into the gas-liquid chromatograph, determine the response ratio for each injection, and average the resulting ratios for each solution. Divide the average response ratio for each solution by the corresponding p,p'-DDT content (in mg) and compare the resulting response factors. These factors should agree to within 2%. Failure to meet this requirement indicates either a weighing error in the preparation of one of the calibration solutions or instrumental difficulties, which must be corrected before proceeding with the analysis of samples.
(c) Preparation of sample. Weigh (to the nearest 0.1 mg) enough sample to contain about 200 mg of pure p,p'-DDT (*w* mg) into a 50 ml stoppered conical flask equipped with teflon-lined screw cap (to estimate the amount of sample which must be taken, consider the nominal percentage of technical DDT for the

formulation assume 70% p,p'-DDT in the technical material). To the flask add by pipette 5 ml of internal standard solution and 20 ml of chloroform. Cap the flask tightly, and gently swirl the contents for 1 minute using a rotational motion of the wrist. Allow the flask to stand at least 5 minutes and then shake for 1 minute, either by hand or using a reciprocal shaker. Ensure that no leaking occurs around the cap. Allow the flask to stand for 30 minutes.
(d) Determination. Inject duplicate aliquots of appropriate volume (as determined in section *(b)*) of the calibration solution B. The response ratios should agree to within 2%. If this precision limit is not met, inject two more aliquots of the solution. Failure to meet the precision requirement with the second pair of injections indicates instrumental difficulties, which must be resolved before proceeding with the analyses. Then inject duplicate aliquots of the sample solution, using the same volume as that used in the preceding step. The precision considerations discussed in the preceding step apply here also. Average the response ratios for each sample solution *(R)*. In a series of analyses, after every two sample solution injections, inject duplicate aliquots of calibration solution B. Average the response ratios of the calibration solution *(R')* injections immediately before and after the sample solution injections. Use this average to calculate the p,p'-DDT content of the sample.

(e) Calculation

$$\text{p,p'-DDT content} = \frac{R \times s \times P}{R' \times w}$$

where:

R	=	average area ratio of p,p'-DDT to the internal standard for the sample solution
R'	=	average area ratio of p,p'-DDT to the internal standard for the calibration solution
s	=	mass of p,p'-DDT in calibration solution (mg)
w	=	mass of sample taken (mg)
P	=	purity of the standard p,p'-DDT (g/kg)

Repeatability r = 23.5 g/kg at 750 g/kg active ingredient content

Reproducibility R = 24.4 g/kg at 750 g/kg active ingredient content

Based on results of 9 participants and 18 values.

p,p'-DDT EMULSIFIABLE CONCENTRATES
*3/TC/M/-

1 Sampling. Take at least 500 ml.

2 Identity test. As for p,p'-DDT technical **3**/TC/M/2.

3 p,p'-DDT. As for p,p'-DDT technical **3**/TC/M/3.

Repeatability r = 3.6 g/kg at 190 g/kg active ingredient content

Reproducibility R = 11.2 g/kg at 190 g/kg active ingredient content

Based on results of 8 participants and 16 values.

p,p'-DDT WETTABLE POWDERS
*3/WP/M/-

1 Sampling. Take at least 500 g.

2 Identity test. As for p,p'-DDT technical **3**/TC/M/2.

3 p,p'-DDT. As for p,p'-DDT technical **3**/TC/M/3, except :
(c) Preparation of sample. Weigh (to the nearest 0.1 mg) enough sample to contain about 200 mg of pure p,p'-DDT (*w* mg) into a 50 ml stoppered conical flask equipped with teflon-lined screw cap (to estimate the amount of sample which must be taken, consider the nominal percentage of technical DDT for the formulation and assume 70% p,p'-DDT in the technical material). To the flask add by pipette 5 ml of internal standard solution and 20 ml of chloroform. Cap each flask tightly and gently swirl the contents for 1 minute using a rotational motion of the wrist. Allow the flask to stand at least 5 minutes and then shake for 1 minute, either by hand or using a reciprocal shaker. Ensure that no leaking occurs around the cap. Allow the flask to stand for 30 minutes. Take a 10 ml aliquot and centrifuge or filter before injection. Continue according to **3**/TC/M/3 *(c)* beginning at: "Then inject aliquots of appropriate volume.... etc.".

* CIPAC-WHO method 1990. Prepared by Mr F.C. Churchill, enters for Disease Control, Atlanta, USA.

Repeatability r = 15.7 g/kg at 548 g/kg active ingredient content

Reproducibility R = 22.7 g/kg at 548 g/kg active ingredient content

Based on results of 9 participants and 18 values.

4 Suspensibility

REAGENTS AND APPARATUS As for MT 15, CIPAC F, p. 45 and for **3**/TC/M/3

PROCEDURE

(a) Preparation of suspension MT 15.1 (*i*)
(b) Determination of sedimentation MT 15.1 (*ii*)
(c) Determination of p,p'-DDT in the bottom 25 ml of suspension. Transfer the sediment quantitatively with water into a tared large evaporating dish.
Evaporate the water by heating on a boiling water bath. Remove the dish as soon as the last traces of water have evaporated. Dry in an oven at 100 °C for 15 min. Cool and reweigh. Transfer the sample to a stoppered conical flask (50 ml) equipped with a teflon-lined screw cap. Add by pipette internal standard solution (5 ml) and chloroform (20 ml) and continue according to **3**/TC/M/3 (*c*).
(*d) Calculation*

$$\text{Suspensibility} = \frac{111(c - Q)}{c} \%$$

where :

c = mass of p,p'-DDT in the sample taken for the preparation of the suspension (g)
Q = mass of p,p'-DDT in the bottom 25 ml of suspension (g)

p,p'-DDT DUSTABLE POWDERS
[*]**3**/DP/M/-

1 Sampling. Take at least 500 g.

2 Identity test. As for p,p'-DDT technical 3/TC/M/2.

3 p,p'-DDT. As for p,p'-DDT technical **3**/TC/M/3, except:

[*] CIPAC-WHO method 1990. Prepared by Mr F.C. Churchill, enters for Disease Control, Atlanta, USA.

(c) Preparation of sample. Weigh (to the nearest 0.1 mg) enough sample to contain about 200 mg of pure p,p'-DDT (*w* mg) into a 50 ml stoppered conical flask equipped with teflon-lined screw cap (to estimate the amount of sample which must be taken, consider the nominal percentage of technical DDT for the formulation and assume 70% p,p'-DDT in the technical material). To the flask add by pipette 5 ml of internal standard solution and 20 ml of chloroform. Cap the flask tightly and gently swirl the contents for 1 minute using a rotational motion of the wrist. Allow the flask to stand at least 5 minutes and then shake for 1 minute, either by hand or using a reciprocal shaker. Ensure that no leaking occurs around the cap. Allow the flask to stand for 30 minutes. Take a 10 ml aliquot and centrifuge or filter before injection. Continue according to **3**/TC/M/3 *(c)* beginning at: "Then inject aliquots of appropriate volume .... etc".

Repeatability r = 2.0 g/kg at 81 g/kg active ingredient content

Reproducibility R = 5.6 g/kg at 81 g/kg active ingredient content

Based on results of 9 participants and 18 values.