TEST METHOD

Effective: 2 April, 1986 DOWM 100024
Supersedes: (New)

o-Benzyl-p-chlorophenol

1. Scope

This method is applicable to the assay of o-benzyl-p-chlorophenol (OBCP). It has been validated over the concentration range of 90 - 100%.

2. Principle

A solution of OBCP containing an internal standard is injected into a capillary gas chromatograph and the components are detected with a flame ionization detector. Quantitation is made by peak area measurement using internal standardization and a computing integrator.

3. Safety

3.1 Each analyst should be acquainted with the potential hazards of the equipment, reagents, products, solvents, and procedures before beginning laboratory work. SOURCES OF INFORMATION INCLUDE: OPERATION MANUALS, MATERIAL SAFETY DATA SHEETS, LITERATURE AND OTHER RELATED DATA. Safety information for products should be requested from the supplier. Disposal of waste materials, reagents, reactants, and solvents must be in compliance with laws and regulations from all applicable governmental agencies.

3.2 o-Benzyl-p-chlorophenol (OBCP) and o-phenylphenol (OPP) are irritants. Avoid skin or eye contact.

3.3 p-Xylene is a flammable solvent. Work in a well-ventilated area away from any source of ignition.

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4. Interferences

No direct interferences have been observed in the use of this method. If results are suspect based on the analytical history of the product, the data should be confirmed by an alternate method.

5. Apparatus

5.1 Capillary gas chromatograph, Hewlett-Packard Model 5890 equipped with a flame ionization detector, or equivalent. The GC must be capable of three stage temperature programming. The 5890 is available from Hewlett-Packard 39550 Orchard Drive, Novi, MI 48050.

5.2 Capillary column, 15 m x 0.53 mm coated with DB-1 (methyl silicone) liquid phase, 1.5 micron film thickness available from J & W Scientific, Inc., 3871 Security Park Drive, Rancho Cordova, CA 95670.

5.3 Syringe, ten microliter: Hamilton 701N, or equivalent, available from Fisher Scientific Company, Midland, MI 48640.

5.4 Computing integrator: Hewlett-Packard 3392A, capable of interfacing to Model 5890 GC, available from Hewlett-Packard, 39550 Orchard Hill Drive, Novi, MI 48050.

5.5 Analytical balance, capable of measuring 0.1 mg: Mettler Model AE163, available from Mettler Instrument Corp., Princeton-Hightstown Road, Hightstown NJ 08520.

5.6 Adapter-liner for on-column injection with megabore size columns, manufactured by a glass fabrication firm according to the specifications in Figure 1. The liner should be inserted in the split injection port in the same manner as a split liner is. Insert the column into the injection port, making sure the front of the column is inserted to the restriction. During an injection, the syringe needle will actually be inserted inside the column.

5.7 Oxygen trap for carrier gas: Go-Getter brand, available from the Anspec Co., 122 Enterprise Drive, P.O. Box 2044, Ann Arbor, MI 48107.

6. Reagents

6.1 p-Xylene, distilled-in-glass grade, available from Fisher Scientific Company, Midland, MI 48640.

6.2 o-Benzyl-p-chlorophenol, available from the DOWICIDE* Antimicrobial Products Coordinator, 2040 Dow Center, The Dow Chemical Company, Midland, MI 48674.

6.3 2-Phenylphenol (OPP), 99+% GOLD LABEL, available from Aldrich Chemical Co., Inc., 940 W. St. Paul Ave., Milwaukee, WI 53233.

These reagents should be checked for purity under the conditions used in the procedure and, if interfering compounds are present, adjustments must be made in preparing the standard.

7. Instrumental Conditions

Capillary Chromatographic Conditions:

Instrument:	Hewlett-Packard 5890
Column:	15 m x 0.53mm DB-1, 1.5 micron film thickness, J & W Scientific Inc.
Temperatures	
Oven:	Three Stage Ramp:
	33°C/1 min to 150°C/1 min at 35°/min
	150°C to 210°C/0.1 min at 10°/min
	210°C to 280°C/4 min at 25°/min
Injection port:	150 °C
Detector:	300 °C
Carrier Gas:	Helium, 13 mL/min at 4 psig
Air flow rate:	250 mL/min
Hydrogen flow rate:	25 mL/min
Make up flow rate:	12 mL/min
Sample size:	1 microliter
Range:	8
Attenuation:	0

See Figure 2 for a typical chromatogram and computing integrator parameters.

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8. Preparation of Standard and Calibration

8.1 Internal standard solution preparation: Weigh 2.0 g of OPP to the nearest 0.1 mg into a 200-mL volumetric flask and dilute to the mark with p-xylene. Mix thoroughly and transfer to an 8-oz glass bottle and seal with a polyethylene-lined cap.

8.2 Weigh 0.1 g of OBCP standard to the nearest 0.1 mg into a 4-dram vial. Into the same vial, add a 10-mL aliquot of internal standard solution using a glass pipette, close with a polyethylene-lined cap, and mix thoroughly.

8.3 Using glass pipets, take a 1-mL aliquot of the solution prepared in 8.2 and add to a 2-dram vial. Dilute with a 4-mL aliquot of p-xylene, close with a polyethylene-lined cap, and mix thoroughly.

8.4 Make a 1-microliter injection of the standard into the chromatograph. Separate according to the chromatographic conditions in Section 7.

8.5 Calibrate the computing integrator according to instrument manufacturer's operating instructions for internal standard methods (See Figure 1 for suggested parameters.)

8.6 Make duplicate injections of the standard into the chromatograph and separate according to the chromatographic conditions in Section 7. Response factors should agree to within the relative precision of the method. If they do not, recalibration should be made.

8.5 If manual calculations are used, calculate the response factor for each component as follows:

$$RF = \frac{A \times B}{C \times D}$$

where:

RF = response factor
A = peak area of internal standard
B = g of component of interest in standard
C = peak area for component of interest in standard
D = g of internal standard in standard mixture

9. Procedure

9.1 Weigh 0.1 g of OBCP sample to the nearest 0.1 mg into a 4 dram vial. Into the same vial add a 10-mL aliquot of internal standard solution using a glass pipette, close with a polyethylene-lined cap, and mix thoroughly. Then dilute according to the procedure in section 8.3.

9.2 Enter internal standard and sample weight into the integrator.

9.3 Make a 1 microliter injection of the sample into the chromatograph and separate according to the chromatographic conditions in Section 7.

10. Calculations

If manual calculations are used, calculate the amount of each component present as follows:

$$\% \text{ OBCP} = \text{RF} \times \frac{E \times F}{G \times H} \times 100$$

where:

RF = response factor for OBCP calculated in Section 8.
E = peak area for OBCP in sample
F = g of internal standard added to sample
G = peak area of internal standard in sample
H = g of sample

11. Precision

Data obtained by this procedure indicate a relative standard deviation of 0.6%. The values obtained may be expected to vary from the average by not more than 1.2% relative at the 95% confidence level.

12. Recovery

Analysis of a series of synthetic mixtures gave recoveries that averaged 99.6% with a range of 98.3- 100.8% and a relative standard deviation of 0.9%.

13. Linearity

Detector response was found to be linear for both the assay component and the internal standard from one-half to twice the concentrations measured.

14. Notes

14.1 The standard solution should be stored in a refrigerator when not in use.

14.2 The purity of the standards should be determined prior to use. The calibration solution should then be corrected for any of these impurities that are present.

14.3 Calibration should be redetermined with every series of unknowns to compensate for variations in the FID output and chromatographic condition changes.

Figure 1. Heated On-Column Adapter-Liner for Megabore Columns

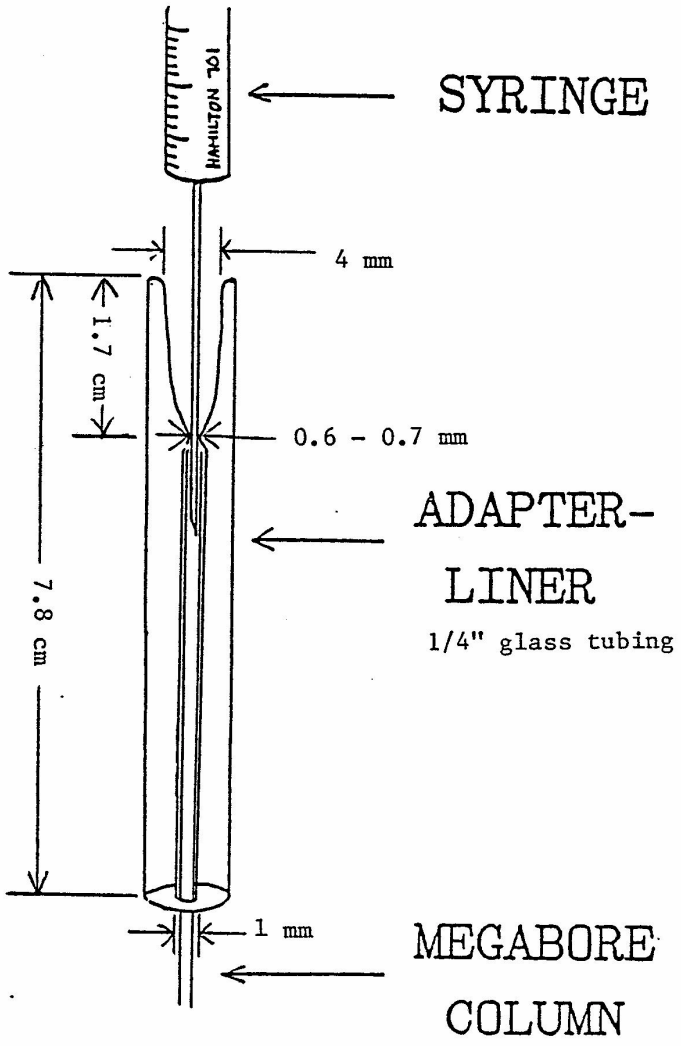
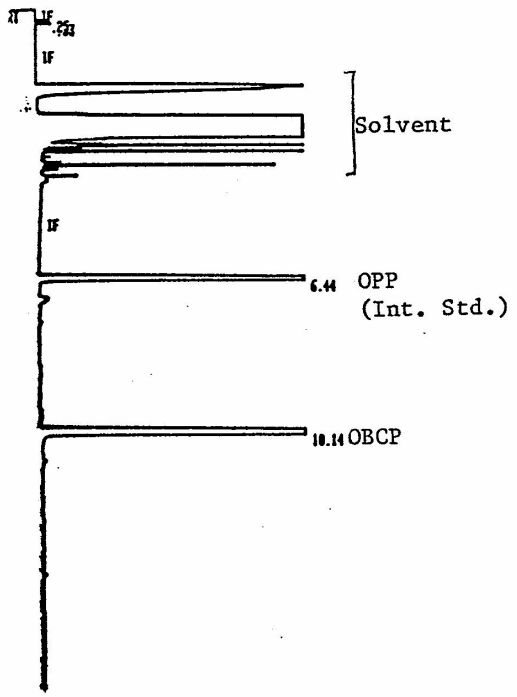


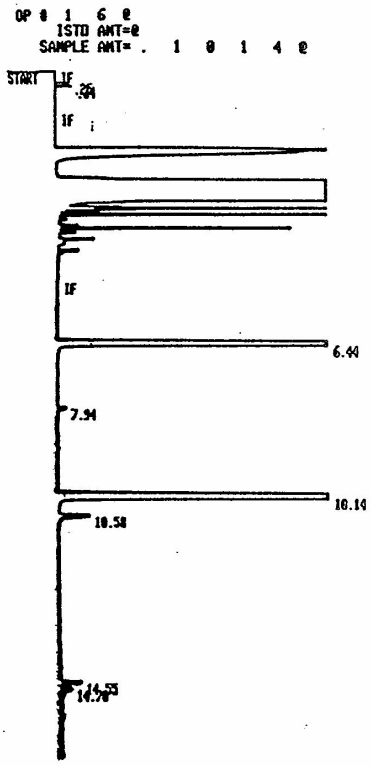
Figure 2. Sample Chromatograms of Standard and Sample of o-Benzyl-p-chlorophenol

Standard




```
:UN # 657
:ORKFILE ID: B5
:ORKFILE NAME:

:REA%
   RT        AREA TYPE   AR/HT     AREA%
 0.33         977   PB   0.010     0.048
 6.44     1099000   PB   0.062    54.101
10.14      931420   PB   0.082    45.850

:OTAL AREA=    2031400
:UL FACTOR= 1.0000E+00


:ALIB  ISTD

:EF % RTW=  5
   % RTW=  5

:AL #       RT          AMT
 1 = 6  .  4  4   =  .  1  0  0
1  2
 2 = 1  0  .  1  4
                  =  .  1  0  2
7
 3 =

:EF PK CAL # 1
:EF PK CAL #

ISTD CAL #  1

LIST: CALIB
ISTD                CALIB RUNS  1
:EF % RTW=  5.00    % RTW=  5.00

CAL #     RT        AMT       AMT/AREA
  1%    6.44   1.0012E-01   9.1098E-08
  2    10.14   1.0270E-01   1.1026E-07
```

Sample




```
OP # 1  6  0
   ISTD AMT=0
 SAMPLE AMT=  .  1  0  1  4  0

RUN # 658
WORKFILE ID: B5
WORKFILE NAME:

ISTD
   RT        AREA TYPE  CAL #     AMOUNT
 6.44     1106100   PB    1%      98.738
10.14      894960   PB    2       96.697

TOTAL AREA=    2001000
ISTD AMT= 1.0012E-01
SAMPLE AMT= 1.0140E-01
NUL FACTOR= 1.0000E+00
```

Typical Integrator Parameters:

```
RUN PRMTRS
ZERO    =  10
ATT 2?  =  2
CHT SP  =  1.0
PK WD   =  0.01
THRSH   =  2
NR REJ  =  500
```