

SPECIFICATIONS AND TESTING PROCEDURE

Product Name	2,3-DICHLORO BENZOYL CYANIDE		
Product Code	DBC	Reference	IN-HOUSE
STP No.	SC/IN/DBC/ST1/00	CAS No.	77668-42-9
Spec. No.	SC/IN/DBC/001/00	Page No.	Page 1 of 17

Details of the material:

Chemical Name	2,3-DICHLORO BENZOYL CYANIDE
CAS N	77668-42-9
Molecular weight	200.03 g/mol
Molecular formula	C ₈ H ₃ Cl ₂ NO
Sampling quantity	10 g
Storage condition	Store in well-closed containers at controlled room Temperature.

Specifications Details:

S. No.	Test Parameters	Specification Limit
1	Description	A off white to pale brown coloured powder.
2	Solubility	Soluble in methanol, chloroform.
3	Identification	
	By IR	The infrared spectrum of the sample is concordant with the spectrum obtained from the similar determination of 2,3 Dichloro benzoyl cyanide Test standard.
	By HPLC	The retention time of the major peak in the chromatogram of the sample solution should

	Prepared By	Reviewed By	Approved By
Name			
Sign and Date			

SPECIFICATIONS AND TESTING PROCEDURE

Product Name		2,3-DICHLORO BENZOYL CYANIDE		
Product Code		DBC	Reference	IN-HOUSE
STP No.		SC/IN/DBC/ST1/00	CAS No.	77668-42-9
Spec. No.		SC/IN/DBC/001/00	Page No.	Page 2 of 17
S. No.	Test Parameters		Specification Limit	
			correspond to that of standard solution as obtained in the test of Assay by HPLC.	
4	Water by Karl Fischer method		Not more than 1.00% w/w	
5	Related substances (By HPLC)			
	2,6 Dichloro Benzoyl Cyanide		Not more than 0.10%	
	2 Chloro Benzoyl Cyanide		Not more than 0.10%	
	2,5 Dichloro Benzoyl Cyanide		Not more than 0.10%	
	2,4 Dichloro Benzoyl Cyanide		Not more than 0.10%	
	3 Chloro Benzoyl Cyanide		Not more than 0.10%	
	4 Chloro Benzoyl Cyanide		Not more than 0.10%	
	3,4 Dichloro Benzoyl Cyanide		Not more than 0.10%	
	3,5 Dichloro Benzoyl Cyanide		Not more than 0.10%	
	Unspecified impurity		Not more than 0.30%	
	Total impurities		Not more than 1.0%	
6	Assay (By HPLC)		2,3 Dichloro benzoyl cyanide contains Not less than 98.0% w/w and not more than 102.0% w/w of C ₈ H ₃ Cl ₂ NO	

	Prepared By	Reviewed By	Approved By
Name			
Sign and Date			

SPECIFICATIONS AND TESTING PROCEDURE

Product Name	2,3-DICHLORO BENZOYL CYANIDE		
Product Code	DBC	Reference	IN-HOUSE
STP No.	SC/IN/DBC/ST1/00	CAS No.	77668-42-9
Spec. No.	SC/IN/DBC/001/00	Page No.	Page 3 of 17

Test Procedure:**1. Description**

Take about 1.0 g of sample in a clean and dry watch glass placed on a sheet of white paper under a white light environment and observe the appearance, color and presence of any other foreign particles.

2. Solubility

Soluble in methanol (1g in 30 ml)

Soluble in chloroform (1g in 30 ml)

3. Identification**A) By IR:**

Examine the sample in potassium bromide dispersion and compare with the spectrum obtained from similar preparation of 2,3 Dichloro benzoyl cyanide Test standard.

OR

Examine the sample as such using UATR technique and compare with the spectrum obtained by the same technique 2, 3 Dichloro benzoyl cyanide Test standard.

B) By HPLC:

Compare the retention time of the major peak in the chromatogram of the sample solution to that of the retention time of the major peak in the chromatogram of standard solution as obtained in the test of Assay.

	Prepared By	Reviewed By	Approved By
Name			
Sign and Date			

SPECIFICATIONS AND TESTING PROCEDURE

Product Name	2,3-DICHLORO BENZOYL CYANIDE		
Product Code	DBC	Reference	IN-HOUSE
STP No.	SC/IN/DBC/ST1/00	CAS No.	77668-42-9
Spec. No.	SC/IN/DBC/001/00	Page No.	Page 4 of 17

4. Water:

Equipment: Karl Fischer titrator (MI-450) or Equivalent

Procedure: Transfer enough methanol to the titration vessel, ensuring that the volume is sufficient to cover the electrodes (approximately 30 to 40 mL) and titrate with the reagent to the electrometric endpoint to consume any moisture that may be present. (Discard the volume consumed since it does not enter the calculation). Quickly add 1g of test sample, mix and again titrate with the reagent to the electrometric or visual endpoint. Calculate and record the Perform the test in duplicate Titrations of the sample and Calculate by the formula.

$$\text{Titre value} \times \text{water equivalence of KF} \times 100$$

$$\text{Water content- (\%, w/w)} = \frac{\text{-----}}{1000 \times \text{weight of the sample(g)}}$$

5. RELATED SUBSTANCES (By HPLC):

Solution A: Weigh 10.0g Sodium Dihydrogen Phosphate Monohydrate in 1000 mL Purified water (conductance value should be less than 0.1µε). add 5.0 ml Butanol. Adjust pH 3.0 ± 0.02 with Ortho Phosphoric Acid.

Solution B: Methanol

Solution C: Acetonitrile (use gradient grade)

Chromatographic Conditions:

Standard Column Type: C18, 4.6 mm x 5cm, 2.7µm

Suitable column: Supelco Ascentis Express, 4.6 mmx 5 cm, 2.7 µm

	Prepared By	Reviewed By	Approved By
Name			
Sign and Date			

SPECIFICATIONS AND TESTING PROCEDURE

Product Name	2,3-DICHLORO BENZOYL CYANIDE		
Product Code	DBC	Reference	IN-HOUSE
STP No.	SC/IN/DBC/ST1/00	CAS No.	77668-42-9
Spec. No.	SC/IN/DBC/001/00	Page No.	Page 5 of 17

Detector: UV

Detector parameter: at wavelength 230nm

Flow rate: 2.0 ml per min

Injection volume :5µL

Column oven temperature: 45°C

Mode: Gradient program

Time (in min)	Solution A (%)	Solution B (%)	Solution C (%)
0.01	85.0	7.5	7.5
4.00	85.0	7.5	7.5
6.00	70.0	10.0	20.0
9.50	70.0	10.0	20.0
10.00	85.0	7.5	7.5
14.00	85.0	7.5	7.5

Retention time principal peak: About 2.9 minutes

Syringe rinse diluent: Diluent

Diluent: Solution A: Solution C (50:50) v/v

Blank: Diluent

Preparation of System suitability solution:

	Prepared By	Reviewed By	Approved By
Name			
Sign and Date			

SPECIFICATIONS AND TESTING PROCEDURE

Product Name	2,3-DICHLORO BENZOYL CYANIDE		
Product Code	DBC	Reference	IN-HOUSE
STP No.	SC/IN/DBC/ST1/00	CAS No.	77668-42-9
Spec. No.	SC/IN/DBC/001/00	Page No.	Page 6 of 17

Weigh accurately about 25 mg of each 2,6 Dichloro Benzoic Acid, 2 Chloro Benzoic Acid, 2,3 Dichloro Benzoic Acid, 2,5 Dichloro Benzoic Acid, 2,4 Dichloro Benzoic Acid, 3 Chloro Benzoic Acid, 4 Chloro Benzoic acid, 3,4 Dichloro Benzoic Acid, 3,5 Dichloro Benzoic Acid and transfer into 25ml volumetric flask. Add 15-20 ml of diluent. Sonicate to dissolve and dilute up to the mark with diluent. Further dilute 1.0ml of above solution to 100.0 ml with diluent. Further dilute 10.0ml of above solution to 100.0 ml with diluent.

Preparation of Standard solution:

Weigh accurately about 25 mg of 2, 3 Dichloro Benzoic acid Test standard and transfer in 25 ml volumetric flask. Add 15-20 ml of diluent. Sonicate to dissolve and dilute up to the mark with diluent. Further dilute 1.0 ml of above solution to 100.0 ml with diluent. Further dilute 10.0ml of above solution to 100.0 ml with diluent.

Preparation of Sample solution:

Weigh accurately and transfer about 25mg of sample in 25 ml volumetric flask. Add 15-18 ml of diluent. Sonicate to dissolve and dilute up to mark with diluents

Note: Prepare Standard solution and Sample solution in duplicate.

Procedure:

Separately inject equal volumes of solutions as per Sequence of injection into the chromatograph

and record the peak area responses for the major peaks and check for the System suitability requirements. Disregard the peak area due to blank interference.

	Prepared By	Reviewed By	Approved By
Name			
Sign and Date			

SPECIFICATIONS AND TESTING PROCEDURE

Product Name	2,3-DICHLORO BENZOYL CYANIDE		
Product Code	DBC	Reference	IN-HOUSE
STP No.	SC/IN/DBC/ST1/00	CAS No.	77668-42-9
Spec. No.	SC/IN/DBC/001/00	Page No.	Page 7 of 17

Sequence of injection:

- 1) Blank
- 2) System suitability solution
- 3) Blank
- 4) Standard solution A_{1.....6}
- 5) Standard solution B
- 6) Blank
- 7) Sample solution A
- 8) Sample solution B
- 9) Blank
- 10) System suitability solution
- 11) Blank
- 12) Standard solution A

System suitability requirements:

The test is not valid unless,

- 1) The Resolution between the peaks corresponding to 2,3 Dichloro benzoic acid and 2,5 Dichloro benzoic acid and between any other two peaks obtained from the injection of System suitability solution is not less than 2.0 and 1.2 respectively. This resolution to be checked on System suitability solution injected at the initial and end of the sequence. (Report the value of the system suitability solution injected at the initial and the end of the sequence)

	Prepared By	Reviewed By	Approved By
Name			
Sign and Date			

SPECIFICATIONS AND TESTING PROCEDURE

Product Name	2,3-DICHLORO BENZOYL CYANIDE		
Product Code	DBC	Reference	IN-HOUSE
STP No.	SC/IN/DBC/ST1/00	CAS No.	77668-42-9
Spec. No.	SC/IN/DBC/001/00	Page No.	Page 8 of 17

- 2) The relative standard deviation for the peak area response of 2,3 Dichloro benzoic acid for replicate injections of Standard solution A and replicate injections of Standard solution A including bracketing standard is not more than 5.0% each and Relative standard deviation for retention time for the peak of 2,3 Dichloro benzoic acid for replicate injections of Standard solution A and replicate injections of Standard solution A including bracketing Standard solution A is not more than 1.0% and 2.0% respectively.
- 3) The Similarity factor between Standard solution A and Standard solution B (separately prepared and injected) is between 0.95 and 1.05. Calculate similarity factor using following formula:

$$\frac{\text{Average peak area response of 2,3 Dichloro Benzoic acid obtained with standard solution A}}{\text{Peak area response of 2,3 Dichloro benzoic acid Obtained with standard solution B}} \times \frac{\text{weight of 2,3 Dichloro benzoic acid Standard in standard solution B in mg}}{\text{Weight of 2,3 dichloro benzoic acid Standard in standard solution A in mg}}$$

Note: If the Similarity factor does not fall within 0.95 to 1.05, prepare fresh Standard solution in duplicate, re-inject in single injection and calculate Similarity factor again as above. If the similarity factor falls within the limit, inject the re-prepared Standard solution A in replicate and. continue the sequence for Blank and sample.

	Prepared By	Reviewed By	Approved By
Name			
Sign and Date			

SPECIFICATIONS AND TESTING PROCEDURE

Product Name	2,3-DICHLORO BENZOYL CYANIDE		
Product Code	DBC	Reference	IN-HOUSE
STP No.	SC/IN/DBC/ST1/00	CAS No.	77668-42-9
Spec. No.	SC/IN/DBC/001/00	Page No.	Page 9 of 17

Approximate Relative Response factor (RRF) and Retention time (RT) for known impurities is given below:

Content	RRF	RT	RRT
2,6 Dichloro Benzoic Acid	1.36	0.59	0.20
2, Chloro Benzoic Acid	0.82	1.58	0.54
2,3 Dichloro Benzoic Acid	---	2.90	1.0
2,5 Dichloro Benzoic Acid	0.45	3.4	1.17
2,4 Dichloro Benzoic Acid	0.57	5.5	1.90
3 Chloro Benzoic Acid	0.41	5.96	2.06
4 Chloro Benzoic Acid	0.31	6.20	2.14
3,4 Dichloro Benzoic Acid	0.48	8.35	2.88
3,5 Dichloro Benzoic Acid	0.68	8.65	2.98

Calculations:

% Content of 2,6 Dichloro Benzoyl Cyanide

$$= \frac{A \times W1 \times 1 \times 10 \times 25 \times 100 \times 200.03 \times 1.36}{J \times 25 \times 100 \times 100 \times W2 \times 191.01}$$

	Prepared By	Reviewed By	Approved By
Name			
Sign and Date			

SPECIFICATIONS AND TESTING PROCEDURE

Product Name	2,3-DICHLORO BENZOYL CYANIDE		
Product Code	DBC	Reference	IN-HOUSE
STP No.	SC/IN/DBC/ST1/00	CAS No.	77668-42-9
Spec. No.	SC/IN/DBC/001/00	Page No.	Page 10 of 17

% Content of 2 chloro benzoyl cyanide

$$\text{Bx W1 x 1x10x25 x100x 165.58 x 0.82}$$

= -----

$$\text{J x 25 x100 x 100 x W2 x 156.57}$$

% Content of 2,5 Dichloro benzoyl cyanide

$$\text{C x W1 x 1x10x25 x100x 200.03 x 0.45}$$

= -----

$$\text{J x 25 x100 x 100x W2 x 191.01}$$

% Content of 2,4 Dichloro benzoyl cyanide

$$\text{D x W1 x 1x10x25 x100x 200.03 x 0.57}$$

= -----

$$\text{J x 25 x100 x 100x W2 x 191.01}$$

% Content of 3 Chloro benzoyl cyanide

$$\text{E x W1 x 1x10x25 x100x 165.58 x 0.41}$$

= -----

$$\text{J x 25 x100 x 100x W2 x 156.57}$$

	Prepared By	Reviewed By	Approved By
Name			
Sign and Date			

SPECIFICATIONS AND TESTING PROCEDURE

Product Name	2,3-DICHLORO BENZOYL CYANIDE		
Product Code	DBC	Reference	IN-HOUSE
STP No.	SC/IN/DBC/ST1/00	CAS No.	77668-42-9
Spec. No.	SC/IN/DBC/001/00	Page No.	Page 11 of 17

% Content of 4 Chloro benzoyl cyanide

$$F \times W1 \times 1x10x25 \times 100x 165.58 \times 0.31$$

= -----

$$J \times 25 \times 100 \times 100x W2 \times 156.57$$

% Content of 3,4 Dichloro benzoyl cyanide

$$G \times W1 \times 1x10x25 \times 100x 200.03x 0.48$$

= -----

$$J \times 25 \times 100 \times 100x W2 \times 191.01$$

% Content of 3,5 Dichloro benzoyl cyanide

$$G \times W1 \times 1x10x25 \times 100x 200.03x 0.68$$

= -----

$$J \times 25 \times 100 \times 100x W2 \times 191.01$$

% Unspecified impurity

$$J \times W1 \times 1x10x25 \times 100x 200.03$$

= -----

$$J \times 25 \times 100 \times 100x W2 \times 191.01$$

% Total unknown impurities = % Sum of all unknown impurities

% Total impurities = % Sum of all known impurities + % Total unknown impurities

Where,

200.03- Molecular Wt. of Dichloro Benzoyl Cyanide

	Prepared By	Reviewed By	Approved By
Name			
Sign and Date			

SPECIFICATIONS AND TESTING PROCEDURE

Product Name	2,3-DICHLORO BENZOYL CYANIDE		
Product Code	DBC	Reference	IN-HOUSE
STP No.	SC/IN/DBC/ST1/00	CAS No.	77668-42-9
Spec. No.	SC/IN/DBC/001/00	Page No.	Page 12 of 17

191.01-Molecular Wt. of Dichloro Benzoic Acid

165.58 - Molecular Wt. of Monochloro Benzoyl Cyanide

156.57-Molecular Wt. of Monochloro Benzoic Acid

A = Peak area response of 2,6 Dichloro Benzoyl Cyanide obtained in the chromatogram of Sample solution.

B = Peak area response of 2 Chloro Benzoyl Cyanide obtained in the chromatogram of Sample solution.

C = Peak area response of 2,5 Dichloro benzoyl cyanide obtained in the chromatogram of sample solution

D = Peak area response of 2,4 Dichloro benzoyl cyanide obtained in the chromatogram of sample solution

E = Peak area response of 3 Chloro benzoyl cyanide obtained in the chromatogram of sample solution

F = Peak area response of 4 Chloro benzoyl cyanide obtained in the chromatogram of sample solution

G = Peak area response of 3,4 Dichloro benzoyl cyanide obtained in the chromatogram of sample solution

H = Peak area response of 3,5 Dichloro benzoyl cyanide obtained in the chromatogram of sample solution

I = Peak area response of unspecified impurity obtained in the chromatogram of sample solution

J = Average peak area response of 2,3 Dichloro benzoic acid obtained in the chromatogram of standard solution A.

	Prepared By	Reviewed By	Approved By
Name			
Sign and Date			

SPECIFICATIONS AND TESTING PROCEDURE

Product Name	2,3-DICHLORO BENZOYL CYANIDE		
Product Code	DBC	Reference	IN-HOUSE
STP No.	SC/IN/DBC/ST1/00	CAS No.	77668-42-9
Spec. No.	SC/IN/DBC/001/00	Page No.	Page 13 of 17

W1 = Weight of 2,3 Dichloro Benzoic Acid Standard in Reference solution A in (g)

W2 = Weight of sample in (g)

Limit of detection & limit of quantitation with respect to sample are as follows:

Impurities	Limit of detection (%)	Limit of quantitation (%)
For known impurities	0.01	0.04
Unspecified impurity	0.01	0.04

Reporting:

1. If the values of the impurity is below detection limit, then report as “Below LOD (LOD value in %)”.
2. If the values of the impurity is more than or equal to LOD and less than LOQ, the report as “Below LOQ (LOQ value in %)”.
3. If no peak is observed at the expected retention time of specified impurity, then report as “Not detected”.

6. Assay (By HPLC)

Preparation of buffer: Dissolve 7.7 g of Ammonium acetate in 1000 mL of water. Adjust the pH to 3.8 ± 0.02 with acetic acid.

Preparation of mobile phase: Prepare a filtered and degassed mixture of 800 mL of buffer and 200 mL of Acetonitrile

	Prepared By	Reviewed By	Approved By
Name			
Sign and Date			

SPECIFICATIONS AND TESTING PROCEDURE

Product Name	2,3-DICHLORO BENZOYL CYANIDE		
Product Code	DBC	Reference	IN-HOUSE
STP No.	SC/IN/DBC/ST1/00	CAS No.	77668-42-9
Spec. No.	SC/IN/DBC/001/00	Page No.	Page 14 of 17

Chromatographic conditions

Standard column Type: C18 Column (25 cm x 4.6 mm, 5 µm)

Suitable column : Inertsil ODS 3V

Detector : UV

Detector parameter : At wavelength 230 nm

Flow rate : 2.0 mL per minutes

Injection volume : 10µl

Column oven Temperature : 35°C

Mode : Isocratic

Run Time : 10 Minutes

Retention time of principal peak : About 4.0 minutes

Syringe rinse diluent : Diluent

Diluent : Mix 500 mL of water and 500 mL of Acetonitrile

Preparation of standard solution:

Weigh accurately about 50.0 mg of the 2,3 Dichloro Benzoyl cyanide standard in a 50 mL Volumetric flask. Dissolve and dilute to volume with diluent.

Preparation of Sample solution:

	Prepared By	Reviewed By	Approved By
Name			
Sign and Date			

SPECIFICATIONS AND TESTING PROCEDURE

Product Name	2,3-DICHLORO BENZOYL CYANIDE		
Product Code	DBC	Reference	IN-HOUSE
STP No.	SC/IN/DBC/ST1/00	CAS No.	77668-42-9
Spec. No.	SC/IN/DBC/001/00	Page No.	Page 15 of 17

Weigh accurately about 50.0 mg of the sample in a 50 mL volumetric flask. Dissolve and dilute to volume with diluent.

Note: Prepare standard solution and sample solution in duplicate.

Procedure:

Separately inject equal volumes of solutions as per Sequence of injection into the chromatograph and record the peak area responses for the major peaks and check for the System suitability requirements.

Sequence of injections:

1. Blank
2. Standard solution A₁.....5
3. Standard solution B
4. Blank
5. Sample solution A
6. Sample solution B
7. Standard solution A

System suitability requirements:

The test is not valid unless,

1. The relative standard deviation for the peak area response of 2,3-Dichloro benzoyl cyanide for replicate injections of standard solution A and replicate injections of standard solution A including bracketing standard is not more than 2.0% each, and relative standard deviation for

	Prepared By	Reviewed By	Approved By
Name			
Sign and Date			

SPECIFICATIONS AND TESTING PROCEDURE

Product Name	2,3-DICHLORO BENZOYL CYANIDE		
Product Code	DBC	Reference	IN-HOUSE
STP No.	SC/IN/DBC/ST1/00	CAS No.	77668-42-9
Spec. No.	SC/IN/DBC/001/00	Page No.	Page 16 of 17

retention time for the peak of 2,3 Dichloro Benzoyl Cyanide for replicate injections of standard solution A and replicate injections of standard solution A including bracketing standard is not more than 1.0% and 2.0% respectively.

- The similarity factor between standard solution A and standard solution B (separately prepared and injected) is between 0.98 and 1.02. Calculate similarity factor using the following formula:

$$\begin{aligned}
 & \text{Average peak area response of 2,3 Dichloro Benzoyl Cyanide obtained with standard solution A} \\
 & \text{Weight of 2,3 Dichloro Benzoyl Cyanide in standard solution B in mg} \\
 = & \frac{\text{Peak area response of 2,3 Dichloro Benzoyl Cyanide obtained with standard solution B}}{\text{Weight of 2,3 Dichloro Benzoyl Cyanide in standard solution A in mg}} \times \frac{\text{Weight of 2,3 Dichloro Benzoyl Cyanide in standard solution B in mg}}{\text{Weight of 2,3 Dichloro Benzoyl Cyanide in standard solution A in mg}}
 \end{aligned}$$

Note: If the similarity factor does not fall within 0.98 to 1.02, prepare fresh standard solution in duplicate, re-inject in single injection, and calculate the similarity factor again as above. If the similarity factor falls within the limit, inject the re-prepared standard solution A in replicate and continue the sequence for Blank and sample.

$$\begin{aligned}
 \% \text{ Assay} = & \frac{A \times W_1 \times 50 \times 100 \times P}{B \times 50 \times W_2 \times (100 - W) \times 100} \times 100
 \end{aligned}$$

	Prepared By	Reviewed By	Approved By
Name			
Sign and Date			

SPECIFICATIONS AND TESTING PROCEDURE

Product Name	2,3-DICHLORO BENZOYL CYANIDE		
Product Code	DBC	Reference	IN-HOUSE
STP No.	SC/IN/DBC/ST1/00	CAS No.	77668-42-9
Spec. No.	SC/IN/DBC/001/00	Page No.	Page 17 of 17

Calculation:

Where:

- **A** = Peak area response of 2,3-Dichloro benzoyl cyanide obtained in the chromatogram of sample solution
- **B** = Average peak area response of principle peak obtained in the chromatogram of standard solution A
- **W1** = Weight of 2,3 Dichloro Benzoyl Cyanide standard in standard solution A in mg
- **W2** = Weight of sample in mg
- **W** = % w/w water of sample
- **P** = % purity of 2,3-Dichlorobenzoyl cyanide standard on an as-such basis

If the assay values obtained are within the limit, calculate the % variation between both the values using the formula:

$$= 2 \times \frac{(\text{Difference between two assay values})}{(\text{Higher assay value} + \text{lower assay value})} \times 100$$

The assay results are acceptable only if the variation is less than 2.0%. If the acceptable criteria for % variation is met, report the mean assay value using the formula:

$$= \frac{\text{Assay value obtained for sample solution (A)} + \text{Assay value obtained for sample solution (B)}}{2}$$

Report: Report the average value as % w/w.

	Prepared By	Reviewed By	Approved By
Name			
Sign and Date			