Washington State Department of Health

TEST KITS

DOH Water Recreation Program

Presenter

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Water Recreation Program Manager

Common pool test kit types

Liquid DPD colorimetric test

FAS-DPD titration test

Photometric (digital) DPD test

Chemicals used in test kits explained

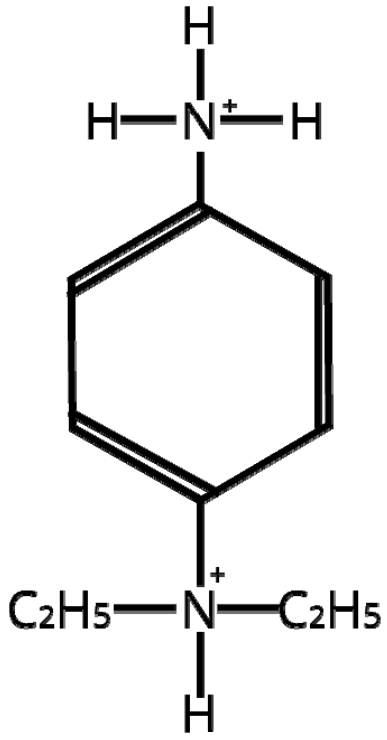
DPD


R-0870
DPD POWDER


KEEP OUT OF REACH OF CHILDREN
R-0002
DPD
REAGENT #2

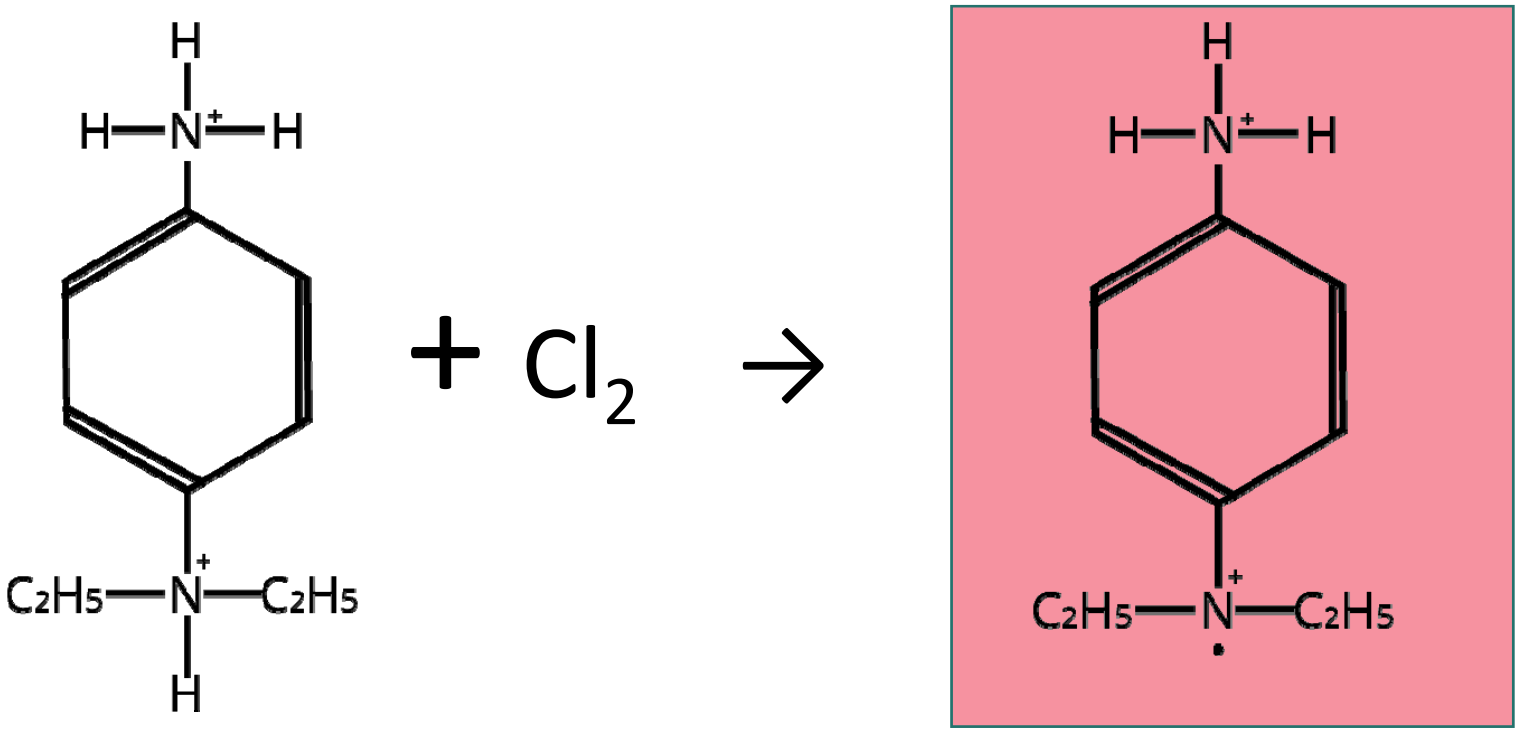
What is DPD?

- DPD: N,N-**d**iethyl-p-**p**henylene **d**iamine
- Can come in solid or liquid form
- Is used in all three below
  - DPD Colorimetric test
  - FAS-DPD titration test
  - Digital colorimetric test



What does DPD do?

In the presence of chlorine, it changes to Würster Dye (red)

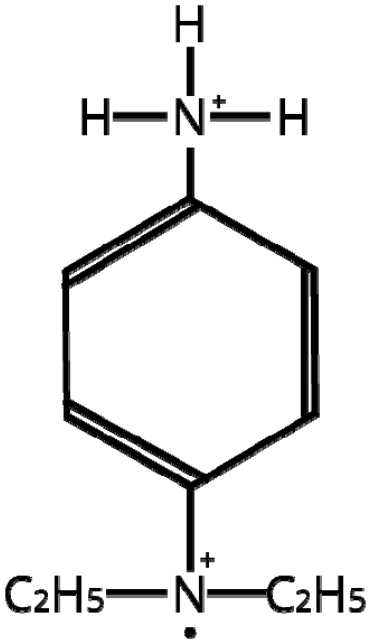


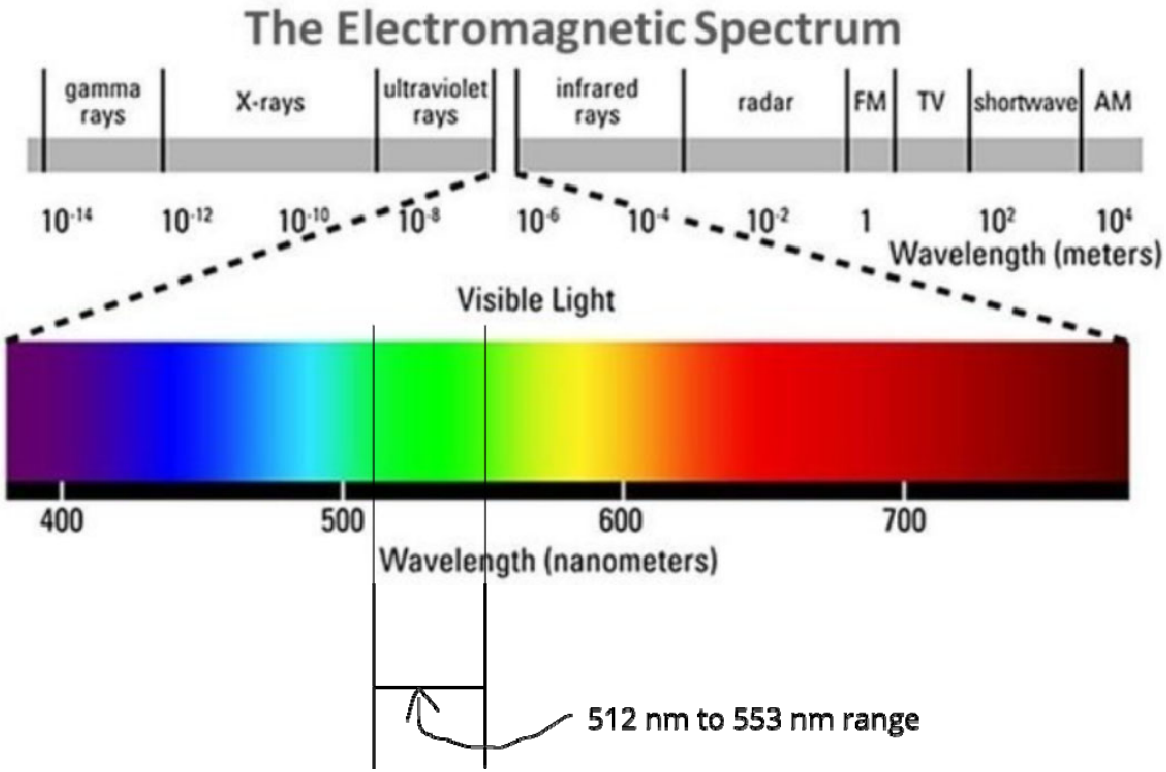
DPD

Würster Dye (red color)

What does DPD do? (continued)

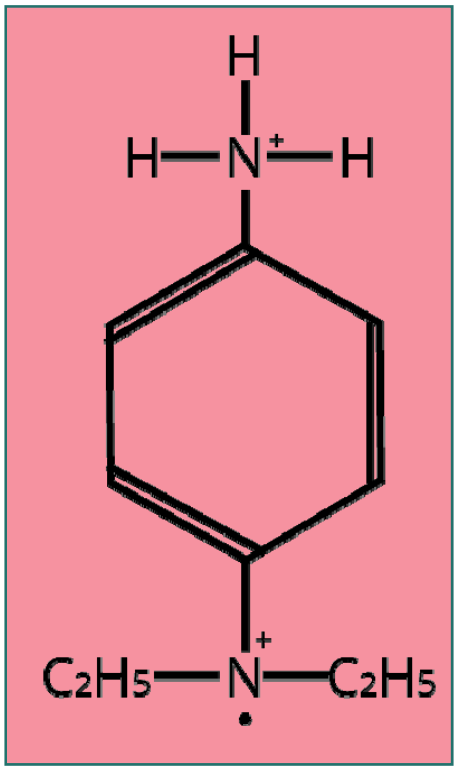
Würster Dye (red) absorbs light (512 nm to 553 nm range)



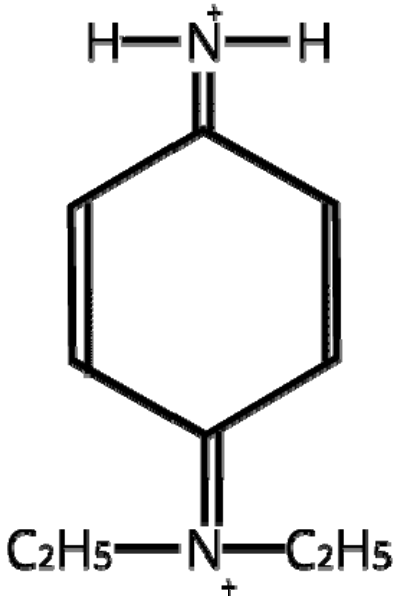


Excess chlorine in sample

Having too much chlorine will **bleach out** the Würster Dye



+ more Cl_2 →



Würster Dye (red color)

Imine (colorless)

What reacts with DPD?

All of below react similarly to DPD to produce red color

- **Cl_2**
- **HOCl**
- **OCl^-**

} Free chlorine by definition

- Chlorine bound to Cyanurate
- Combined chlorines at high concentrations after 1 min
- 50% of NCl_3
- 20% of ClO_2
- Bromine species (HOBr, OBr^-, Bromamines)
- Iodine
- And more

What can interfere with DPD test?

Unwanted factor	Negative effect
Presence of high combined chlorine (>.5ppm)	Shows up as free chlorine after 1 min
Low pH (<6.2)	Combined chlorine shows up as free chlorine
High pH (>8.0)	Dissolved oxygen shows up as free chlorine
Traces of iodide left in test tube	Combined chlorine shows up as free chlorine
Presence of oxidized manganese	Shows up as free chlorine
High free chlorine level (>4.0 ppm)	Non-linearity of DPD colorimetric test (bleaching effect)
High temperature	Combined chlorine shows up as free chlorine

Proper storage and maintenance of DPD

- Store in a tightly capped brown bottle
- Store in the dark
- Store in a cool place
- Discard when discolored
- Discard when past expiration

Potassium Iodide (KI)

What is potassium iodide?

- It is a potassium salt: KI (potassium ion (K^+) and Iodide (I^-))
- Can come in solid or liquid form (usually liquid)
- Used in all three test methods
  - Liquid DPD colorimetric test
  - FAS-DPD titration test
  - Digital colorimetric test

What does potassium iodide do?

- Makes combined chlorines (NH_2Cl and $NHCl_2$) more reactive to DPD
- (with little KI) $NH_2Cl + 3I^-$ (iodide)$+ H_2O + H^+ \rightarrow$

  $NH_4OH + Cl^- + I_3^-$ (triiodide)
- (with more KI) $NHCl_2 + 3I^-$ (iodide)$+ H_2O + 2H^+ \rightarrow$

  $NH_4OH + 2Cl^- + I_3^-$ (triiodide)
- I_3^- (triiodide) reacts with DPD to produce Würster Dye (red color)

What can compromise KI test

Unwanted factor	Negative effect
Oxidation from oxygen	• Iodide gets oxidized to become iodine • Iodine causes DPD to be Würster dye (red) • Falsely high reading in combine/total chlorine test
Oxidation from light	
Loss of iodide through evaporation of iodine	False low combined/total chlorine reading due to depleted iodide in reagent
Presence of potassium monopersulfate	KMPS reacts with Iodide to produce triiodide (False high combined/total chlorine reading)

Proper storage and maintenance of KI

- Store in tightly capped bottle
- Store in the dark
- Store in a cool place
- Discard when yellow
- Discard when past expiration date

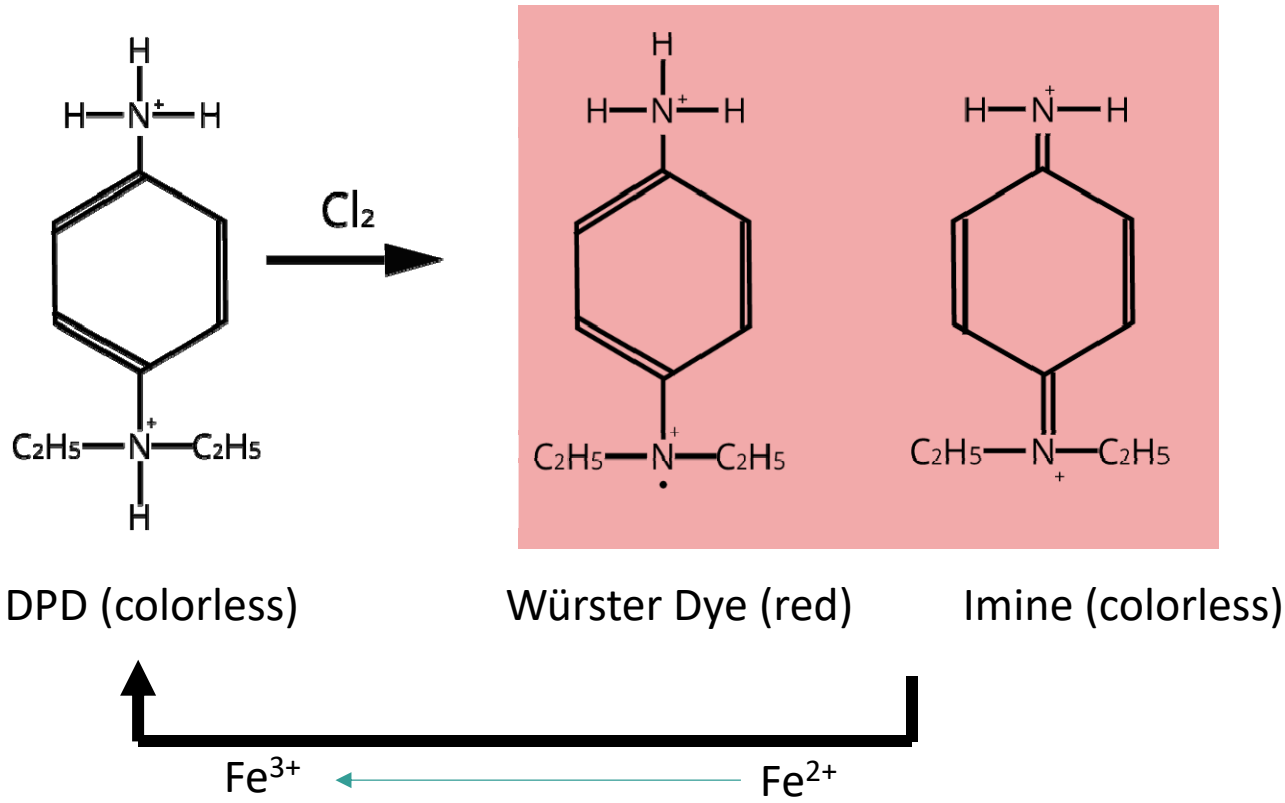
FAS

What is FAS?

- Stands for **f**errous **a**mmonium **s**ulfate
- $[NH_{4+}]_2[Fe^{2+}][SO_4^{2-}]_2$ ammonium/iron salt solution
- Comes in liquid form
- Used in only FAS-DPD titration test

What does FAS do?

Iron [Fe^{2+}] reacts with Würster dye (red)and imine (colorless) to revert them back to DPD (colorless).



What can compromise FAS test

Unwanted factor	Negative effect
Incorrect drop size	
Drop size too big (tilting the bottle)	False low reading
Drop size too small (static electricity around the nozzle)	False high reading
FAS is already oxidized	False high reading

Proper storage and maintenance of FAS

- Store in a tightly capped brown bottle
- Store in the dark
- Store in a cool place
- Discard when past expiration date

DPD colorimetric test procedure

What is really happening? (DPD colorimetric test)

Steps taken		Chemicals introduced	What is happening
1	Fill the tube with sample water	Water Free chlorine (if any) Combined chlorine (if any)	N/A
2	Add the first reagent (pH=10.0)	Phosphoric acid Sodium salt Unidentified chemicals	Conditioning the sample for the next step
3	Add the second reagent (pH=1.3)	DPD Unidentified chemicals	Free chlorine reacts with DPD to produce Würster dye. This is a quick reaction. Do this in less than 1 min to avoid monochloramine showing up.
4	Add the third reagent (pH=7.6)	Potassium iodide Hydriodic acid Unidentified chemicals	KI reacts with combined chlorine to produce triiodide, which reacts with DPD to produce Würster dye. This is a slow reaction (wait 2-3 min)

More complicated procedure for fun

Steps		What is being measured
A	Add 1st and 2nd reagents to fresh sample	Free chlorine
B	Add a small amount (1 drop) of 3rd reagent	Free chlorine Monochloramine
C	Add more (4 drops) of 3rd reagent Wait for 2 minutes	Free chlorine Monochloramine Dichloramine ½ Trichloramine
D	Add a small amount (1 drop) of 3rd reagent to fresh sample. Add 1st and 2nd reagents and read immediately	Free chlorine Monochloramine ½ Trichloramine

Chemical	Reading
Free chlorine	A
Monochloramine	B-A
Dichloramine	C-D
Trichloramine	2(D-B)

FAS-DPD titration test procedure

What is really happening? (FAS-DPD titration test)

Steps		Chemicals introduced	What is happening
1	Fill the tube with sample water	Water Free chlorine (if any) Combined chlorine (if any)	N/A
2	Add DPD powder (pH=N/A)	DPD sulfate EDTA Phosphate Unidentified chemicals	Conditioning the sample water and adding DPD at the same time. Free chlorine reacts with DPD to produce Würster dye.
3	Add FAS titrant drop wise (pH=2.2)	Ferrous ammonium sulfate Unidentified chemicals	FAS reacts with Würster dye to revert it to DPD to lose color. In order to minimize monochloramine interference, do this quickly
4	Add KI (pH=7.6)	Potassium iodide Hydriodic acid Unidentified chemicals	KI reacts with combined chlorine to produce triiodide, which reacts with DPD to produce Würster dye. This is a slow reaction (wait 2-3 min)
5	Repeat step 3. No need to hurry because it is for combined chlorine		

More complicated procedure for fun

Steps		What is being measured
A	Add DPD powder to fresh sample and add FAS titrant until clear	Free chlorine
B	Add one drop of KI and add FAS titrant drop wise until clear	Monochloramine
C	Add 4 more drops of KI and titrate with FAS until clear Wait for 2 min	Dichloramine ½ Trichloramine
D	Add a small amount (1 drop) of KI to fresh sample and DPD powder and titrate rapidly with FAS until clear	Free chlorine Monochloramine ½ Trichloramine

Chemical	Reading
Free chlorine	A
Monochloramine	B
Dichloramine	C
Trichloramine	2(D-(A+B))

Let’s measure chlorine!

Follow the instructions on the handout

- If you are an inspector, find an operator to do testing with
- If you are an operator, find an inspector to do testing with
- Take a sample bottle for each table group
- Measure free chlorine level first
- Measure combined chlorine level next (total chlorine if using liquid DPD reagent)
- Record the readings
- Watch how your partner does testing
- Compare the results with your partner
- Complete this task by 2:05 PM (10 minutes before the end of this presentation)

Cleaning procedure

- **Finish** testing by 2:05 PM (10 minutes before the end of this presentation)
- **Dump** sample water into the "dump bucket"
- **Rinse** your test tube and the sample bottle in the "rinse bucket"
- **Return the sample bottle** to the plastic tub

Questions to check your understanding

- Which type of test kit do you use, Liquid DPD, FAS-DPD, or digital?
- What chemical turns red when chlorine is present?
- What happens when there is too much chlorine?
- What chemical helps combined chlorine to show in the chlorine test?
- Do you need to wait for chemical reaction to complete itself when testing for free chlorine?
- Do you need to wait for chemical reaction to complete itself when testing for combined chlorine?
- Why is correct drop size important, and how can you make sure your drop size is correct?
- What compromises the quality of reagents?

Questions?

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