Bioscience, Inc.
Environmental Products & Services
ISO 9001:2015 Certified

Accu-Test™ Micro Chemical Oxygen Demand System
EPA Approved

Hints for Producing Excellent Results with the Accu-Test™ Micro-COD Method

The micro-COD method, by eliminating reagent mixing, glassware cleaning, wet chemistry analyses, etc., has made the COD test one of the easiest of water quality tests. Since it is a micro technique, it is imperative that each step be followed carefully for good results.

Here are Some Important Hints for Obtaining Consistent Results

Homogenization/Milling

Homogenize or mill your samples. Because of the small sample size, it is important to get a representative sample into the vial. If the sample contains particulates, it is important to mill the samples so that the oxidants have better access to the particle for digestion.

Chlorides

Frequently the chloride level of samples is unknown. An excess of 1000 mg of chloride will tend to make COD analyses (macro or micro) highly scattered. Samples can be tested quickly for chloride concentrations by potentiometric analysis or mercuric nitrate titration. An accurate number is not necessary, only an approximation. Pretesting for chlorides will save time and money.

Sample dilution, to get your sample below 1000 mg/l chlorides, is preferred prior to testing. Sample pre-treatment to complex the chlorides is the next best method. The pre-treatment method is described under "High Chloride Test Method" in the detailed test procedure available from Bioscience, Inc.

Pipetting

It is essential that an accurate 2.5 ml sample (0.5 ml for high range tests) be placed in the vial. If you are using a fixed volume pipette, check the calibration to make certain you are getting an accurate delivery. Class A volumetric pipets are recommended for sample addition to obtain the highest accuracy.

Dilutions

Use Class A volumetric glassware and at least 10 ml of sample per dilution for highest accuracy. Use multiple dilutions for dilution factors greater than 100 rather than a single dilution.

Layering the Sample in the Vial

Pipette the sample **slowly** into the vial. Tilt the vial allowing the sample to run down the side. Do not squirt the sample directly into the reagents. Volatile materials will be lost and acid will spatter unless this practice is observed. The sample should form a "layer" on top of the reagents.

Mixing After Resealing Vial

Wearing eye protection, apron and gloves, shake the vial or the rack containing the vials vigorously for a few seconds. This mixes the reagent and sample layers.

Wiping the Vial

Using the optical technique for analysis requires that each vial be carefully wiped before placing the vessel in the cuvette well.

Avoid Scratching the Vial

If a scratch is noted on the vial, place it in the cuvette well such that the scratch is away from light path. If the vial is too badly scratched, pour the contents into a clean, scratch-free vial or titrate the sample.

Shake the Vials after Removing From Digestion Block

Often during digestion, samples stratify. Agitate the tube rack, then allow the precipitate to settle (see below). Allow approximately 10 minutes for settling.

Do Not Shake the Vials Immediately Prior to Analysis

A precipitate forms in the digested vessel. If you are using optical analysis, it is important that this precipitate settles and is not disturbed prior to placing the vial in the cuvette well.

Run Your Own Standard Curve

For highest accuracy (and regulated or "certified" analyses), you should not use the standard curves provided with these tips. You should prepare your own. Include at least a blank and mid-range standard with each batch of samples.

If COD Analyses Are Running High (Compared To Reflux Titration Method) Check:

(a) The chloride level of the sample
(b) The stratification of the sample after digestion
(c) The cleanliness of the vial (or scratches in the glass)
(d) The pipette calibration
(e) The accuracy of your standards and spectrophotometer
(f) The zero on your spectrophotometer (and zero drift during analysis)
If you still have the problem, then use a homogenizer or blender long enough to strip all volatiles out of the samples. Note the difference due to volatiles in reporting results.

If Cod Analyses Are Running Low (Compared To Reflux Titration Method) Check:

(a) The layering of the sample onto the reagents
(b) The grinding of particulates in the sample so that the oxidizing agents have better access to the oxidizable constituents in the particulates
(c) The calibration of the pipette
(d) The accuracy of the standards
(e) The digestion block temperature
(f) The stratification of the sample after digestion
(g) The zero on your spectrophotometer (and zero drift during analysis)

Precautions for Low Level COD Tests

(a) Use only distilled or highly purified water with less than 0.1 mg/l Total Organic Carbon (TOC), not tap deionized water, to make your standards and to wash your sample container. Avoid use of detergent washing.

(b) Wear plastic gloves and keep laboratory dust to a minimum. Avoid smoking. A single, nearly invisible skin flake, weighing 15 micrograms, could cause an error of up to 100% at the 5 to 10 mg/l COD level.

(c) Read vials, then rotate 90 degrees. Read again, and average the two readings for the best precision.
CAUTION: Avoid scratches/imperfections in the glass.

(d) Use duplicate or triplicate determinations to improve accuracy.

(e) Use only the special low range accu-TEST™ vials for the 5 to 150 mg/l COD range.

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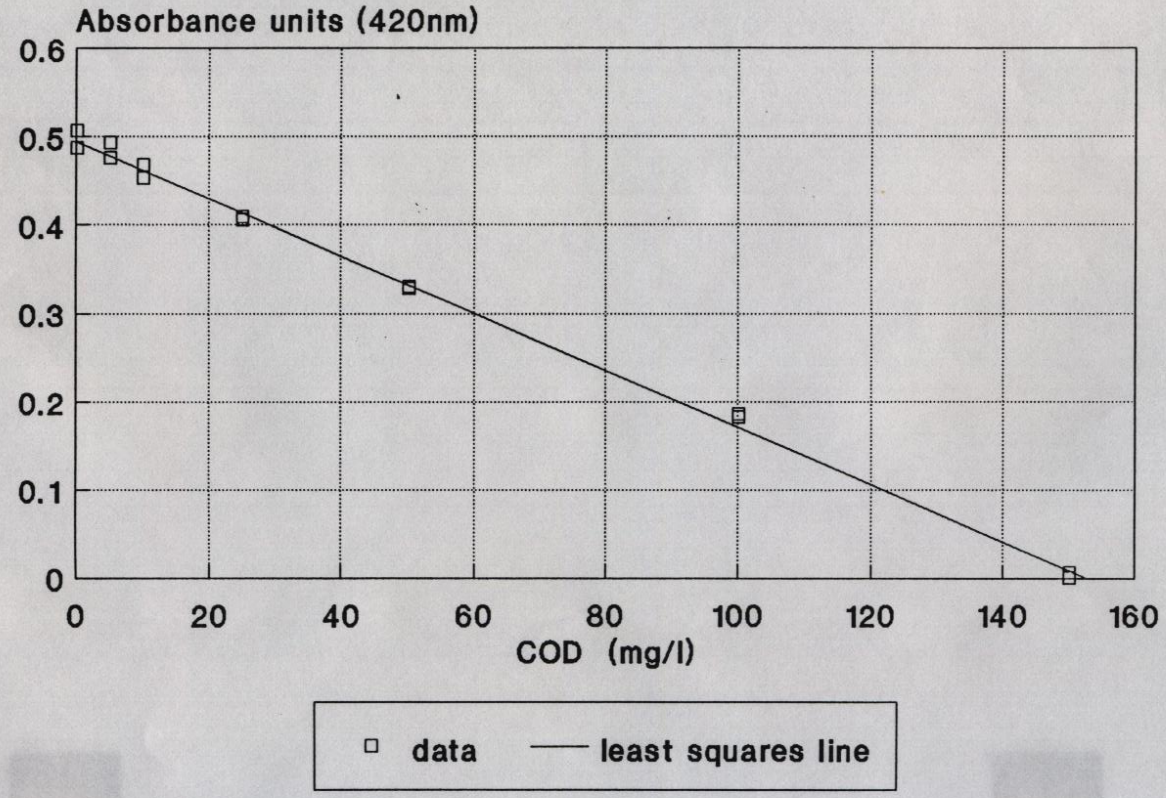
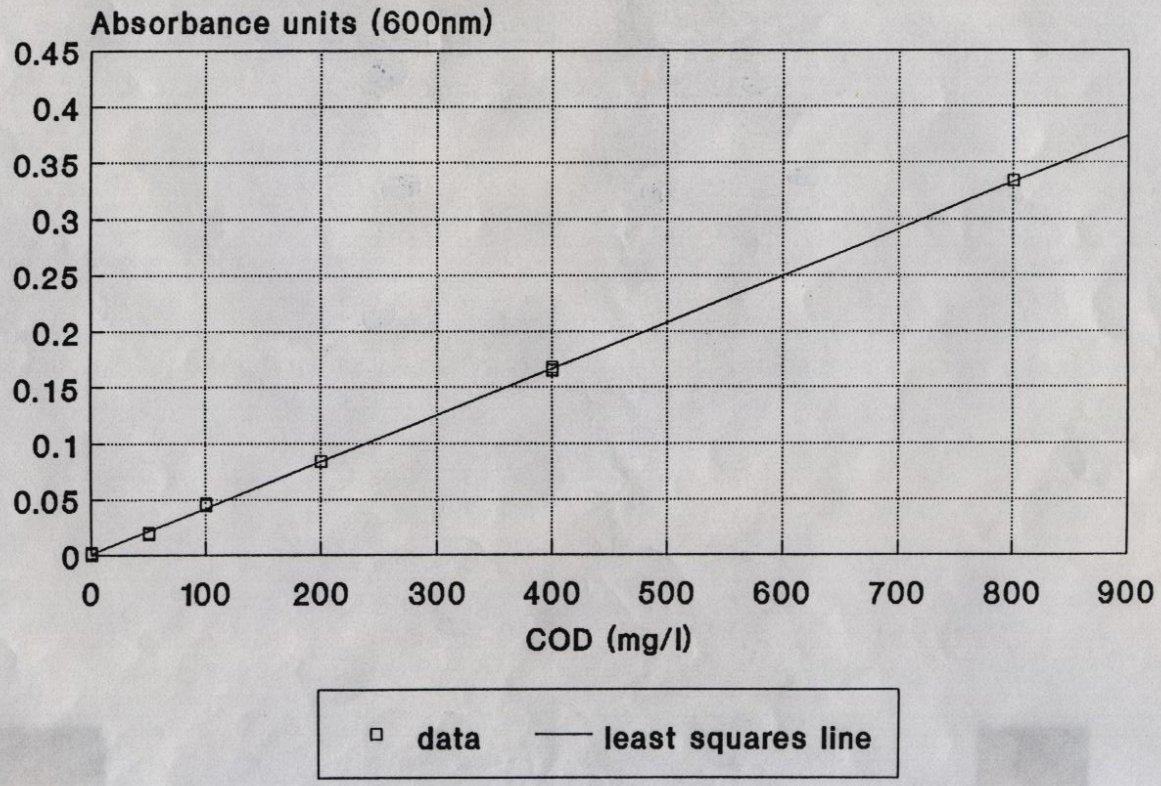
Spectronic 21 COD Calibration Curve
accu-TEST Low Range

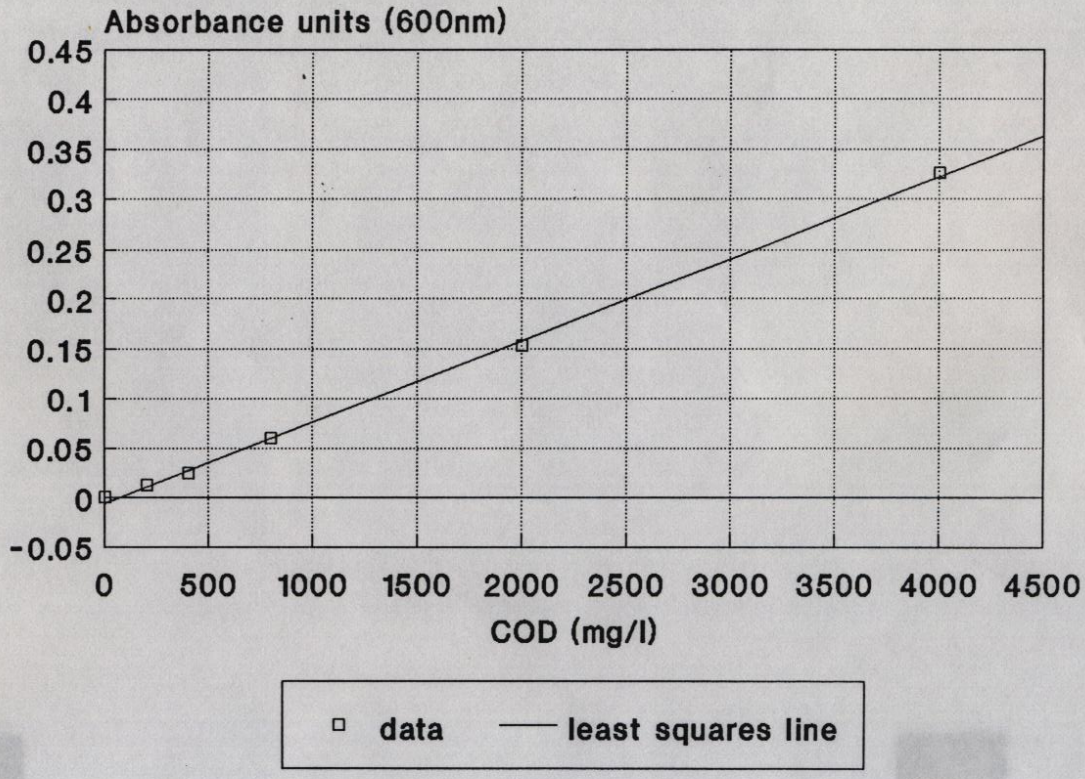


2 ml sample, 420 nm wavelength

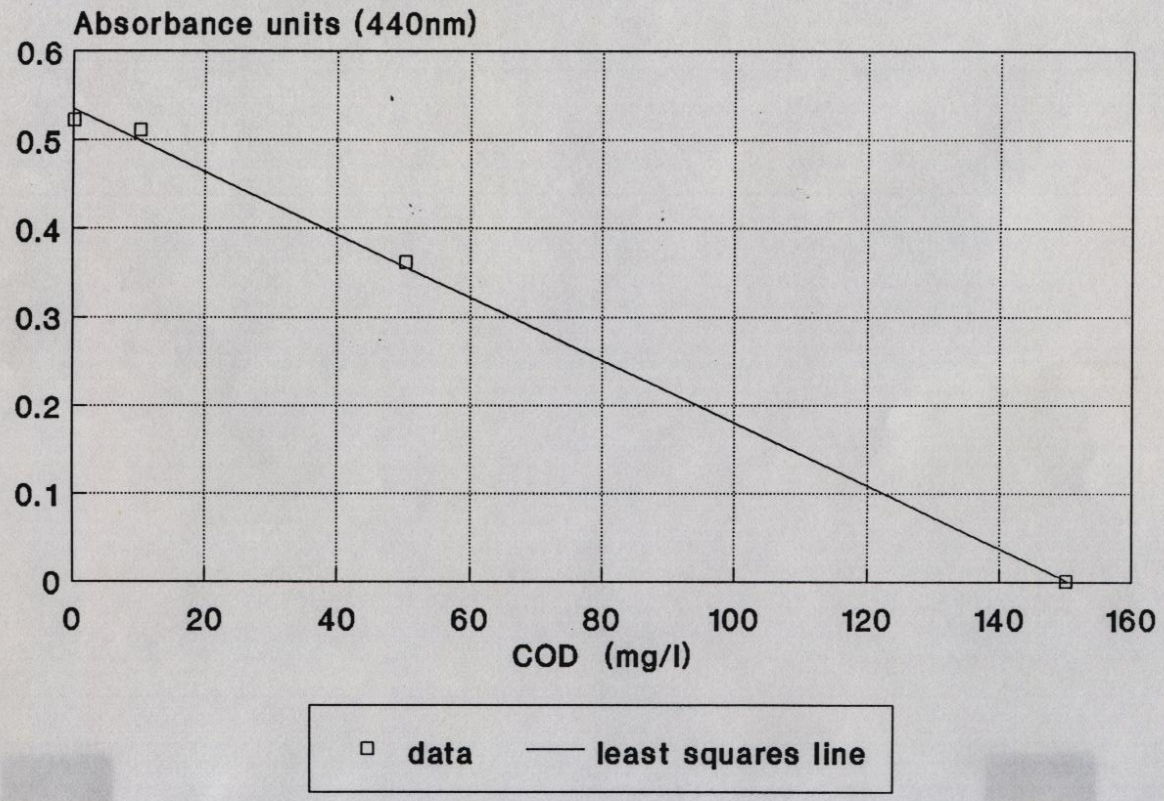
Spectronic 21 COD Calibration Curve
accu-TEST Standard Range



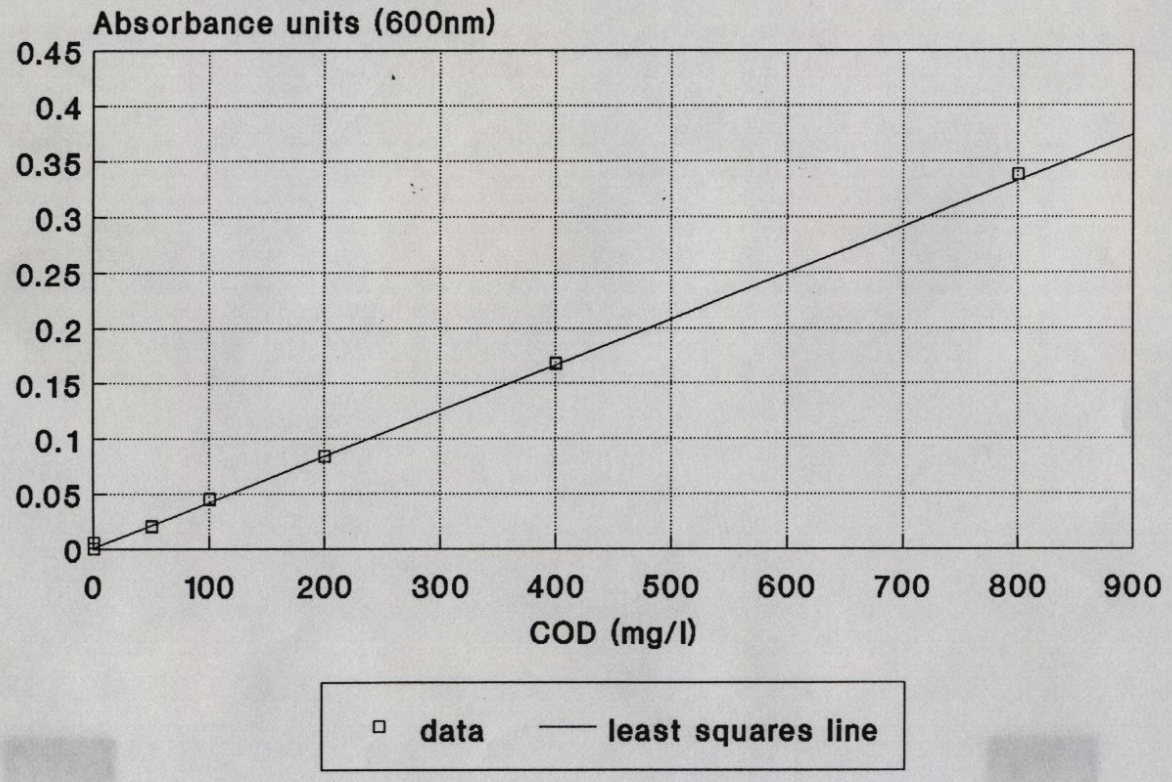
Spectronic 21 COD Calibration Curve
accu-TEST High Range



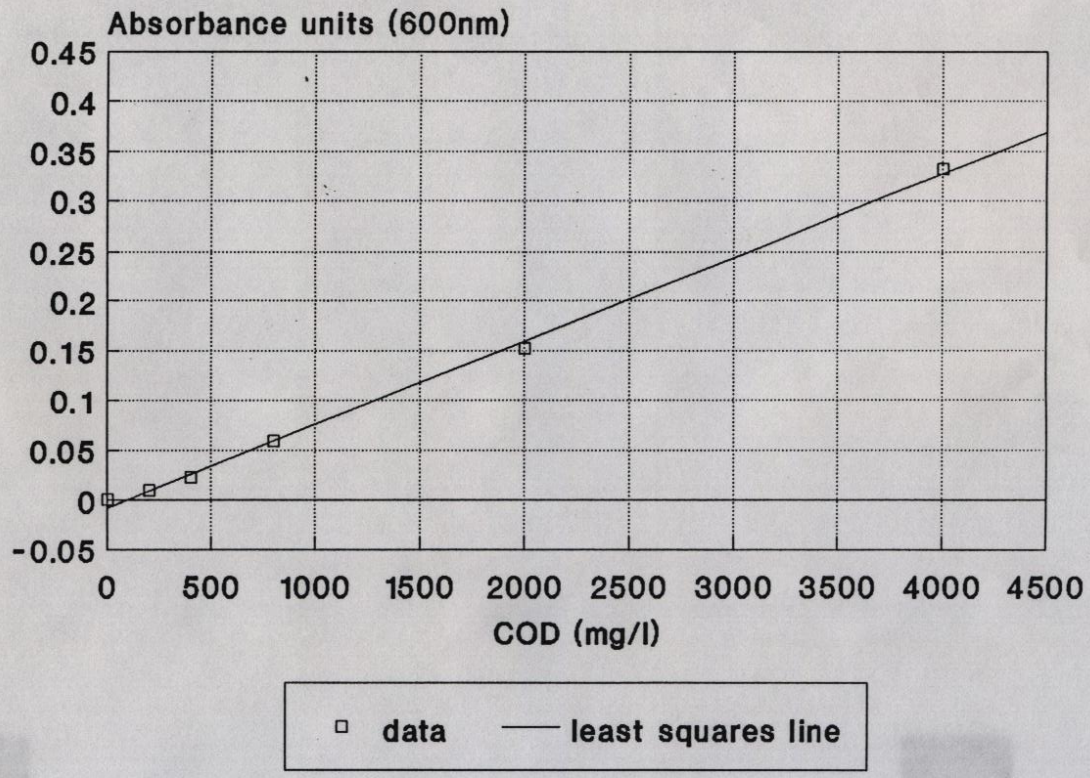
HACH DR3000 COD Calibration Curve
accu-TEST Low Range



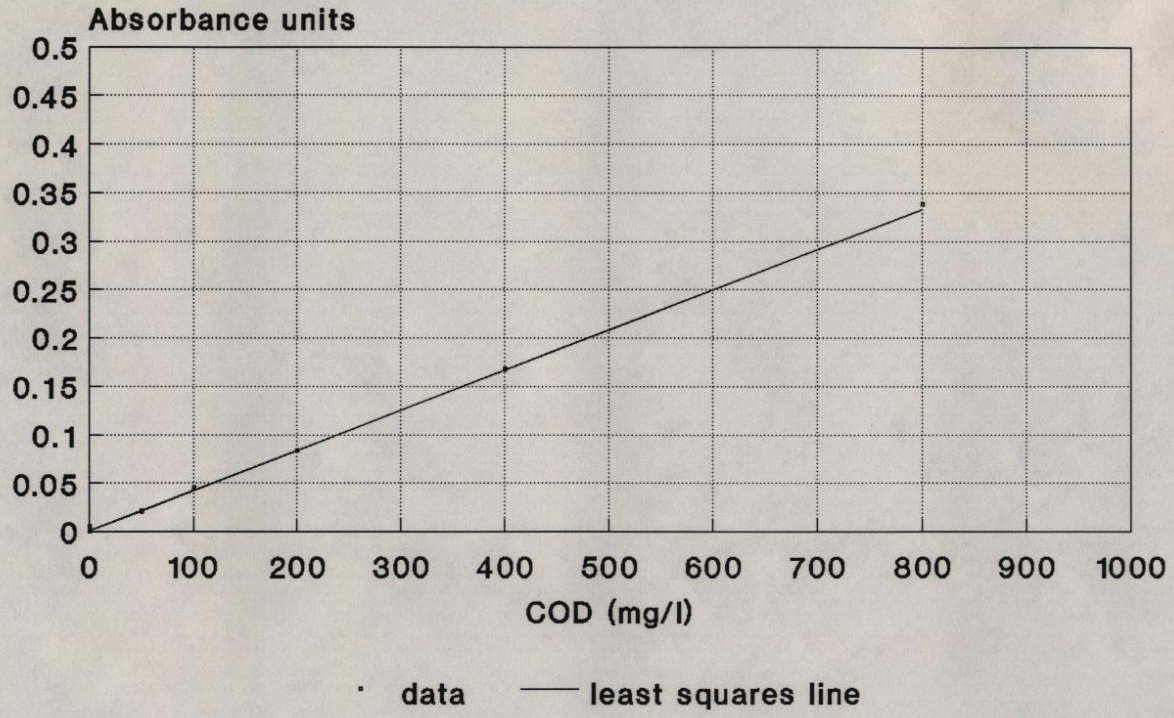
HACH DR3000 COD Calibration Curve
accu-TEST Standard Range



HACH DR3000 COD Calibration Curve
accu-TEST High Range

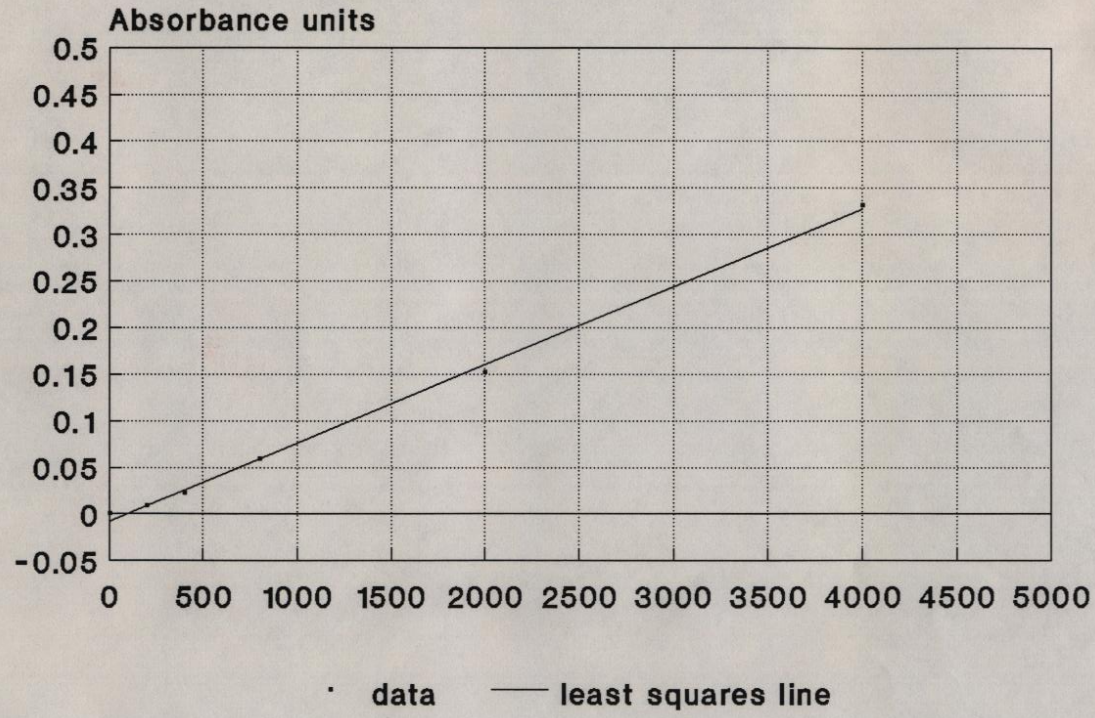


COD Calibration Curve
Bioscience Standard Range



Determined on DR3000

COD Calibration Curve
Bioscience High Range



Determined on DR3000