



ENVIRONMENT AGENCY

An inter-laboratory trial on chemical oxygen demand
determinations (2007)

Methods for the Examination of Waters and Associated Materials

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Methods for the Examination of Waters and Associated Materials

This booklet contains information on an inter-laboratory trial on chemical oxygen demand determinations using some of the methods described in the previous booklet in this series.

Throughout the booklet, the term chemical oxygen demand (COD) is used to express the amount of oxygen consumed during oxidation of a sample with hot acid dichromate solution under defined conditions; the test provides an estimate of the oxidisable matter present in the sample. The result is usually expressed as milligrams of oxygen consumed per litre of sample (mg l^{-1}).

Whilst this booklet may report details of the materials actually used, this does not constitute an endorsement of these products but serves only as an illustrative example. Equivalent products are available and it should be understood that the performance characteristics of the method might differ when other materials are used. It is left to users to evaluate methods in their own laboratories.

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About this series

Introduction

This booklet is part of a series intended to provide authoritative guidance on recommended methods of sampling and analysis for determining the quality of drinking water, ground water, river water and sea water, waste water and effluents as well as sewage sludges, sediments, soil (including contaminated land) and biota. In addition, short reviews of the most important analytical techniques of interest to the water and sewage industries are included.

Performance of methods

Ideally, all methods should be fully evaluated with results from performance tests. These methods should be capable of establishing, within specified or pre-determined and acceptable limits of deviation and detection, whether or not any sample contains concentrations of parameters above those of interest.

For a method to be considered fully evaluated, individual results from at least three laboratories should be reported. The specifications of performance generally relate to maximum tolerable values for total error (random and systematic errors), systematic error (bias), total standard deviation and limit of detection. Often, full evaluation is not possible and only limited performance data may be available.

In addition, good laboratory practice and analytical quality control are essential if satisfactory results are to be achieved.

Standing Committee of Analysts

The preparation of booklets within the series "Methods for the Examination of Waters and Associated Materials"

and their continuing revision is the responsibility of the Standing Committee of Analysts. This committee was established in 1972 by the Department of the Environment and is now managed by the Environment Agency. At present, there are nine working groups, each responsible for one section or aspect of water quality analysis. They are

- 1 General principles of sampling and accuracy of results
- 2 Microbiological methods
- 3 Empirical and physical methods
- 4 Metals and metalloids
- 5 General non-metallic substances
- 6 Organic impurities
- 7 Biological methods
- 8 Biodegradability and inhibition methods
- 9 Radiochemical methods

The actual methods and reviews are produced by smaller panels of experts in the appropriate field, in co-operation with the working group and main committee. The names of those members principally associated with these methods are listed at the back of the booklet.

Publication of new or revised booklets will be notified to the technical press. If users wish to receive copies or advance notice of forthcoming publications, or obtain details of the index of methods then contact the Secretary on the Agency's internet web-site (www.environment-agency.gov.uk/nls) or by post.

Every effort is made to avoid errors appearing in the published text. If, however, any are found, please notify the Secretary.

Dr D Westwood
Secretary
August 2006

Warning to users

The analytical procedures described in this booklet should only be carried out under the proper supervision of competent, trained analysts in properly equipped laboratories.

All possible safety precautions should be followed and appropriate regulatory requirements complied with. This should include compliance with the Health and Safety at Work etc Act 1974 and regulations made under this Act, and the Control of Substances Hazardous to Health Regulations 2002 (SI 2002/2677). Where particular or exceptional hazards exist in carrying out the procedures described in this booklet, then specific attention is

noted. Numerous publications are available giving practical details on first aid and laboratory safety. These should be consulted and be readily accessible to all analysts. Amongst such publications are; "Safe Practices in Chemical Laboratories" and "Hazards in the Chemical Laboratory", 1992, produced by the Royal Society of Chemistry; "Guidelines for Microbiological Safety", 1986, Portland Press, Colchester, produced by Member Societies of the Microbiological Consultative Committee; and "Safety Precautions, Notes for Guidance" produced by the Public Health Laboratory Service. Another useful publication is "Good Laboratory Practice" produced by the Department of Health.

Inter-laboratory trial on chemical oxygen demand determinations

This booklet is based on a DOE funded report prepared by Yorkshire Environmental LabServices.

1 Introduction

1.1 An inter-laboratory study, funded by the Department of the Environment, was set up to test new proposed chemical oxygen demand (COD) methods. These low level ($0\text{--}160 \text{ mg l}^{-1}$) COD small scale open- and closed-tube methods use mercury suppression of chloride interference.

1.2 Results⁽¹⁾ (from three laboratories) indicated that earlier bluebook methods produced by the Standing Committee of Analysts (SCA) using mercury-free methods for the determination of low levels of COD (closed-tube method B and open-tube method C in the 1986 publication⁽²⁾, i.e. methods D and E of the previous booklet) produced positive bias in the presence of low concentrations of chloride. This bias was shown to be very variable. The chloride concentration in many final effluents was found to be sufficient to cause biased results in COD determinations. This bias could be significant with respect to monitoring compliance with the Urban Wastewater Treatment Regulations 1994⁽³⁾.

1.3 Analysts were asked to analyse prepared samples in duplicate over a period of five days using either (or both) procedures described in method B of the previous booklet. In addition, analysts were also requested to compare these methods with any existing methods used in their own laboratories.

1.4 Two previous SCA COD methods⁽²⁾, the 1977 method and the 1986 (method A) method, i.e. methods A and C respectively of the previous booklet, have never been properly tested using an inter-laboratory trial of this sort. It was hoped that using a single set of (16) samples the performance characteristics determined would provide a robust test of the new proposed methods and possibly previously published methods.

1.5 Twelve laboratories participated in the inter-laboratory trial (Table 0). Only six laboratories (marked **) carried out the main part of the trial using the proposed procedures in method B of the previous booklet. Five laboratories used the closed tube variation, and one laboratory used both the closed and the open tube variations.

1.6 Six laboratories used their current in-house methods and/or commercial test kit methods. Five participating laboratories reported results using commercially available closed tube test kits. These test kits measure the light absorption (at 420 nm) of the dichromate ion as a means of determining COD instead of the titration procedure. The sealed digestion tube also acts as a cuvette which allows light absorption measurements to be carried out.

1.7 One laboratory used the original 1977 full scale (10 ml sample volume) mercury-suppressed method⁽⁴⁾, i.e. method A of the previous booklet.

1.8 The types of samples distributed to laboratories are described in Table 2. Table 3 lists the symbols used in the raw data and statistical tables.

1.9 It is important to note that each method was not, in general, carried out by the same number of laboratories, or in some cases, to the same degree of replication. Hence

the simple statistical treatment employed is not completely rigorous, but is considered adequate to indicate the typical performance of the methods.

2 Details of samples circulated

2.1 The pH of all samples, except the one containing humic acid was adjusted to 2.0 ± 0.1 by the addition of 4 M sulphuric acid. The humic acid solution was stabilised with 0.01 M sodium hydroxide to a pH value of 12.

2.2 Participants were asked to commence the analysis as soon as possible after receipt of the samples, but that if this was not possible to store the samples at 4°C before analysis.

2.3 Sewage works final effluent samples were distributed with COD levels ranging from about 40 to 230 mg l⁻¹. Participants were requested to dilute sample 16 with water in the ratio 1:1 prior to analysis..

2.4 The effect of chloride was assessed with synthetic chloride solutions (see Table 2) at concentrations of 500 and 2000 mg l⁻¹ as chloride and with sewage effluents that naturally contained up to about 440 mg l⁻¹ chloride.

2.5 Potassium hydrogen phthalate (KHP) was used to prepare the primary COD standard solutions.

2.6 A complete set of samples was reserved for duplicate analysis to be carried out after 74 days storage at 4°C . The results, expressed as a percentage of the original result are shown in Table 4. The determination was carried out using a commercial test kit in the range 0 - 160 mg l⁻¹.

3 Protocol adopted

3.1 The samples were not identified to the participants. Laboratories were requested to analyse each sample in duplicate over a 5 day period.

3.2 An analysis of variance was carried out⁽⁵⁾ and outliers were rejected (marked # in tables) using Dixon's Q test (two tailed at the 5 % significance level). It is noted that for some laboratories, a result is rejected that may be a consequence of the good precision shown for that laboratory, and not because of the variation shown between laboratories.

3.3 When only one result, instead of the duplicate result requested, was submitted, the result for that day was not included in the statistical treatment, except when the estimated standard deviation was calculated.

3.4 Many laboratories submitted insufficient data for an analysis of variance to be carried out. Consequently, in order to give more information in these cases estimated standard deviations and relative standard deviations were calculated. These values were determined using all the data submitted for a given sample excluding outlier results and results reported as "less than". This procedure was considered useful as a means of comparing the data.

3.5 Table 0 lists the laboratories taking part. Table 1 lists the laboratory and method code numbers; the basis of the methods used by each laboratory; the top COD calibration

standard; the volume of sample used in each determination, and in addition whether mercury suppression was used.

3.6 Used in this study were the closed and open tube versions respectively of method B in the previous booklet. Method D was as an example of the closed tube methods that do not use mercury to suppress chloride interference. The 2-ml sample volume is similar to that of method B of the 1986 reference. Method A is the original 1977 full scale reference SCA method⁽⁴⁾ using 10 ml sample volume and mercury suppression of chloride. Method G was an in-house method with a range of calibration standards between 0 - 1600 mg l⁻¹. Methods F1, F2 and F3 were sealed-tube proprietary test kits that used mercury to suppress chloride interference.

4 Results

4.1 The results for the 16 samples are summarised by sample number in Tables 5 - 20. The first table (for example Table 5A) for each sample shows the raw data, the second table (i.e. Table 5B) for each sample shows the statistical analysis of the data presented in the first table.

4.2 The bias is calculated for standard solutions only where the theoretical COD is known. It is calculated as the difference between the mean result obtained by a given laboratory for a specified method and the theoretical COD of the sample. Table 21 shows the limits of detection calculated for the "effective" blank samples (i.e. the blank, the samples containing 500 and 2000 mg l⁻¹ as chloride respectively) and the lowest COD sample with a theoretical value of 25 mg l⁻¹.

4.3 Although four laboratories reported results for Method D, three of the laboratories reported four or less results per sample, whilst one laboratory reported eight results per sample.

4.4 One laboratory reported no data for method A.

4.5 In addition, results from an experienced analyst were reported for comparison with those reported by an inexperienced analyst (marked * in tables).

5 Effect of chloride

5.1 Overall, insufficient data were provided to ensure that a statistical comparison of results by different methods would be valid.

5.2 However, whilst it is difficult to generalise, it can be seen from Table 22 that in the presence of chloride, the results for method D (the existing mercury-free based method) show higher COD results than methods A, B, or F1 which all used mercury suppression of chloride.

6 Basic performance characteristics of the methods

6.1 Limit of detection

Table 21 shows the limit of detection obtained by the participating laboratories calculated from the results of the blank (sample 9), the 500 and 2000 mg l⁻¹ chloride standard solutions (samples 13 and 3 respectively) and the lowest COD standard of 25 mg l⁻¹ (sample 1).

7 Oxidation of refractory substances

7.1 Table 22 also shows the mean results for the COD standard solutions and the solutions of sodium acetate (nominal COD 128 mg l⁻¹) (sample 5) and humic acid (sample 15) for all methods. The total carbon content of the sodium acetate solution (sample 5) was determined using an OI total organic carbon analyser and found to be within 1% of the theoretical value.

8 Experienced versus inexperienced analysts

8.1 Laboratory 7 submitted results for methods B and F1 using an experienced analyst (two years experience of COD determinations) and an in-experienced graduate analyst (two days training in carrying out COD tests, marked *). These results can be compared by examination of individual tables.

9 Conclusions

9.1 Six of the twelve participating laboratories submitted results for the small scale closed-tube COD procedure of method B of the previous booklet. One laboratory submitted results for the small scale open-tube COD procedure of method B of the previous booklet. Both of these procedures utilise mercury suppression of chloride. The other six laboratories submitted results for their existing methods and/or commercial small scale test kits, also utilising mercury suppression.

9.2 Overall, the precision of the results for method B (open-tube and closed-tube procedures) were similar to method D (closed-tube procedure). However, in the presence of chloride, the results for method D (closed-tube procedure) were in general higher than the corresponding values obtained by method B of the previous booklet. This could have a significant effect with respect to monitoring compliance with the Urban Waste Water Directive⁽³⁾.

10 References

1. Unpublished studies carried out by Yorkshire Environmental, LabServices (Rotherham Laboratory), Thames Water, (Docklands Laboratory) and National Rivers Authority (Leeds Laboratory).
2. Standing Committee of Analysts, Chemical Oxygen Demand (Dichromate Value) of Polluted and Waste Waters, 1986 (Second Edition), *Methods for the Examination of Waters and Associated Materials*, in this series, ISBN 0117519154.
3. The Urban Waste Water Treatment (England and Wales) Regulations 1994, Statutory Instrument 1994/2481.
4. Standing Committee of Analysts, Chemical Oxygen Demand (Dichromate Value) of Polluted and Waste Waters 1977, *Methods for the Examination of Waters and Associated Materials*, in this series, ISBN 0117512494.
5. A Manual of Analytical Quality Control for the Water Industry, Water Research Centre, NS30, R V Cheeseman and A L Wilson, revised by M J Gardner, 1989.

Table 0 Participating laboratories

Analytical and Environmental Services (Wallsend)
Anglian Water Services Limited (Norwich)
DOE (Northern Ireland) Water Executive (East Division) **
Essex Analytical (Hanningfield)
A.H. Marks and Company (Bradford)
North West Water (Warrington) **
National Rivers Authority (Leeds)
National Rivers Authority (Nottingham) **
Severn Trent Laboratories (Coventry) **
South West Water (Exeter)
Thames Water (Docklands) **
Yorkshire Environmental, LabServices (Rotherham) **

Grateful acknowledgement is made to all laboratories which participated in the inter-laboratory trial, and especially those (marked **) that reported results for method B of the previous booklet.

Table 1 **Summary of laboratories and methods**

Laboratory code	Method	Sample volume (ml)	Mercury suppression (Y/N)	Method code in this booklet	COD of top calibration standard (mg/l)
1	Closed-tube procedure of method B	2	Y	B _{C-T}	160
4	Closed-tube procedure of method B	2	Y	B _{C-T}	160
5	Closed-tube procedure of method B	2	Y	B _{C-T}	160
6	Closed-tube procedure of method B	2	X	B _{C-T}	160
7	Closed-tube procedure of method B	2	Y	B _{C-T}	160
8	Closed-tube procedure of method B	2	Y	B _{C-T}	160
7	Open-tube procedure of method B (experienced analyst)	2	Y	B _{O-T}	160
7	Open-tube procedure of method B (in-experienced analyst)	2	Y	B _{O-T*}	160
1	Closed-tube based on SCA 1986 (method D)	10	N	D	400
2	Closed-tube SCA 1986(method D)	2	N	D	400
5	Closed-tube SCA 1986(method D)	2	N	D	400
6	Closed-tube SCA 1986(method D)	2	N	D	400
11	Open-tube SCA 1977 (0 - 400 mg/l) (method A)	10	Y	A	400
3	Laboratory's own method (0 - 1600 mg/l)	2	Y	G	1600
7	Test kit A (0 - 160 mg/l), experienced analyst	2	Y	F1	160
7	Test kit A (0 - 160 mg/l), in-experienced analyst	2	Y	F1*	160
9	Test kit A (0 - 160 mg/l)	2	Y	F1	160
12	Test kit A (0 - 160 mg/l)	2	Y	F1	160
1	Test kit B (0 - 150 mg/l)	2	Y	F2	150
1	Test kit C (0 - 150 mg/l)	2	Y	F3	150
10	Test kit C (0 - 150 mg/l)	2	Y	F3	150

Note: Calibration standard solutions prepared using potassium hydrogen phthalate (KHP). Methods are described in the previous booklet.

Table 2 **Sample identification**

Sample number	Sample	Chloride concentration (mg/l)	Raw data	Table number	Statistical treatment
1	COD (KHP) standard, 25 mg/l.	< 10	6A		6B
2	Sewage final effluent 1	80	14A		14B
3	2000 mg/l chloride standard	2000	13A		13B
4	Sewage final effluent 2	80	15A		15B
5	Sodium acetate; nominal COD 128 mg/l	< 10	9A		9B
6	Sewage final effluent 3	209	16A		16B
7	COD (KHP) standard, 160 mg/l	< 10	8A		8B
8	COD (KHP) standard, 125 mg/l with 500 mg/l chloride	500	12A		12B
9	Blank	< 10	5A		5B
10	Sewage final effluent 4	350	17A		17B
11	COD (KHP) standard, 125 mg/l	< 10	7A		7B
12	Sewage final effluent 5	106	18A		18B
13	500 mg/l chloride	500	11A		11B
14	Sewage final effluent 6	368	19A		19B
15	Humic acid solution	< 10	10A		10B
16	Sewage final effluent 7	440	20A		20B

Table 3 Summary of symbols and abbreviations used in tables

A blank cell indicates that a result was not submitted, or that the result is not relevant, or that insufficient data points are available to calculate the within and between batch standard deviations, or that statistics were unavailable since the between batch mean square is less than the within batch mean square for these data.

#	Indicates that this result has been rejected as an outlier using Dixon's Q test, two tailed, at the 5% significance level; any result obtained on this day is not included in the calculation of within and between batch standard deviations. It is noted that for some laboratories, a result has been rejected probably as a consequence of the excellent precision shown for the batch of results.
Sw	Within batch standard deviation.
St	Total standard deviation.
Sb	Between batch standard deviation.
RSD %	Relative standard deviation as percent.
DoF	Degrees of freedom.
Est SD	Estimated standard deviation. (This is to be compared with the total standard deviation).
Est RSD %	Estimated relative standard deviation as percent.

Results for laboratory 3 (method G) have not been included in combined statistical calculations.

All values are given in mg/l, unless otherwise stated.

Where appropriate, the results in the final three columns of the statistical analysis tables are based on an estimated total standard deviation calculated for all results, except outliers and results reported as "less than".

Table 4**Stability tests performed on samples stored for 74 days at 4 °C**

Sample number	Mean COD value (mg/l) Before storage	Mean COD value (mg/l) After storage
1	24.4	25.5
2	55.4	62.5
3	8	10.5
4	74.7	77.5
5	114.5	118.0
6	111.7	117.5
7	161.2	161.5
8	126.3	128.5
9	1	2.0
10	73.7	82.5
11	127.4	128.0
12	142	126.0
13	2.6	2.5
14	105.6	109.5
15	118.7	121.0
16	228.6	225.0

Table 5A Comparison results for sample 9 (blank, chloride concentration <10 mg/l)

Method code	Laboratory number	Day 1	Day 2	Day 3	Day 4	Day 5
B _{C-T}	1	-6.8	-4.5	-7	-3.2	-7.7
B _{C-T}	4	-4	-4	-4	-4.7	18.1
B _{C-T}	5	8.2	8.2	19.5	14.9	3.6
B _{C-T}	6	2	2	11	3	13
B _{C-T}	7	4	2	1	0.4	1.4
B _{C-T}	8	2.6	6.4	6.5	0.4	7.5
B _{O-T}	7	1	1	0.3	0	0
B _{O-T} *	7	1.9	0.3	-0.7	0.5	0.8
D	1	4	0	-12		
D	2	2	0	-3	-2	0
D	5	8.8		6.6		
D	6	-2	-5	-6	-9	
A	11	2.1	1.1	2	2	-2
G	3	< 5	< 5			
F1	7	4	3	0	0	0
F1*	7	8	4	0	3	8
F1	9	0	4	0	0	0
F1	12	< 5	5			
F2	1	3.3		10	8	
F3	1	< 5	< 5	< 5	< 5	< 5
F3	10	< 10	< 10	< 10	< 10	12.8
					< 10	< 10
					10.8	15.3

Table 5B Statistical analysis of results for sample 9 (blank, chloride concentration <10 mg/l)

Method code	Laboratory number	Mean	Max	Min	Sw	Sb	St	RSD %	DoF	Number of outlier results	Est SD	Number of results used
B _{C-T}	1	-4.74	12.9	-19.6	3.19	9.40	9.93	-209.48	4	0		10
B _{C-T}	4	0.89	18.1	-4.7	2.60	7.39	7.84	880.42	4	0		10
B _{C-T}	5	8.33	19.5	2.6	3.17	4.88	5.82	69.84	5	0		10
B _{C-T}	6	8.00	16.0	2.0	7.36					1	5.8	8
B _{C-T}	7	1.63	4.0	0.4	0.91	0.99	1.35	82.60	5	0		6
B _{C-T}	8	5.76	11.2	0.4	2.93	1.06	3.12	54.17	9	0		10
B _{O-T}	7	0.93	2.0	0.0	0.33	0.83	0.89	95.66	5	0		10
B _{O-T} *	7	-0.39	1.9	-3.0	1.59	0.23	1.60	-413.61	7	0		8
D	1	-2.67	4.0	-12.0						0	8.3	3
D	2	1.25	5.0	-3.0	2.06	2.37	3.14	-251.40	5	0		8
D	5	7.70	8.8	6.6						0	1.6	2
D	6	-5.50	-2.0	-9.0	2.12	2.40	3.20	-58.21	2	0		4
A	11	1.07	2.1	-2.0	1.82					0	1.6	10
G	3	< 5										
F1	7	1.00	4.0	0.0	0.77	1.35	1.56	155.72	5	0		10
F1*	7	2.80	8.0	0.0	3.00	1.15	3.21	114.76	9	0		10
F1	9	1.22	4.0	0.0	2.00					1	1.9	9
F1	12	5.00	5.0	< 5								1
F2	1	7.10	10.0	3.3						0	3.4	3
F3	1	< 5	< 5	< 5								
F3	10	7.39	15.3	< 10							2.3	3

The overall mean of method B is 3.0 ± 3.5 .

Table 6A Comparison of results for sample 1 (KHP COD standard, 25 mg/l)

Method code	Laboratory number	Day 1	Day 2	Day 3	Day 4	Day 5
B _{C-T}	1	39.3	25	24.8	9.4	14.6
B _{C-T}	4	42.8	34.8	29.5	22.3	25.2
B _{C-T}	5	23.8	20.3	36.5	24.1	31.9
B _{C-T}	6	56	30	23	21	27
B _{C-T}	7	26	26	22	25	23
B _{C-T}	8	25.2	25.7	28.8	34.5	32.9
B _{O-T}	7	21_	24	24	26	25
B _{O-T} *	7	32.3	25.9	29	27.5	26.7
D	1	25		30	29	
D	2	25	27	21	23	25
D	5	27.4		23.2		
D	6	46	22	21	15	
A	11	25.6	26	26	26.6	26.7
G	3	120	116			
F1	7	26	18	21	23	28
F1*	7	30	32	27	25	27
F1	9	27	30	21	26	28
F1	12	17	28			
F2	1	23		8.2		14.3
F3	1	< 5	< 5	< 5		26
F3	10	24.4	27.6	18.6	44.2	28.2
					34.6	32.1
					32.1	33.3
						37.8

Table 6B Statistical analysis of results for sample 1 (KHP COD standard, 25 mg/l)

Method code	Laboratory number	Mean	Bias	Max	Min	Sw	Sb	St	RSD %	DoF	Number of outlier results	Est SD	Est RSD %	Number of results used
B _{C-T}	1	20.09	-4.91	39.3	9.4	7.12	6.10	9.37	46.65	7	0			10
B _{C-T}	4	29.01	4.01	42.8	22.3	4.55	4.87	6.67	22.98	8	0			10
B _{C-T}	5	30.31	5.31	42.2	20.3	5.35	3.81	6.56	21.65	7	0			10
B _{C-T}	6	34.10	9.10	56.0	21.0	11.25	5.03	12.32	36.13	8	0			10
B _{C-T}	7	24.50	-0.50	26.0	22.0	1.47	0.82	1.68	6.87	6	0			6
B _{C-T}	8	31.41	6.41	40.3	25.2	3.65	2.68	4.53	14.43	7	0			10
B _{O-T}	7	25.00	0.00	26.0	24.0	0.79					1	0.7	2.8	9
B _{O-T} *	7	26.50	1.50	32.3	22.6	2.76	1.64	3.21	12.12	6	0			8
D	1	28.00	3.00	30.0	25.0						0	2.6	9.4	3
D	2	26.63	1.63	32.0	21.0	2.15	3.40	4.03	15.12	4	0			8
D	5	25.30	0.30	27.4	23.2						0	3.0	11.7	2
D	6	26.00	1.00	46.0	15.0	12.37	7.18	14.30	55.00	2	0			4
A	11	26.06	1.06	27.0	23.7	1.09	0.26	1.12	4.31	9	0			10
G	3	118.00	93.00	120.0	116.0						0	2.8	2.4	2
F1	7	24.40	-0.60	28.0	18.0	2.49					0	3.4	13.8	10
F1*	7	26.50	1.50	32.0	23.0	2.07	2.12	2.96	11.18	6	0			10
F1	9	26.13	1.13	32.0	21.0	3.16	0.67	3.23	12.37	9	0			10
F1	12	22.50	-2.50	28.0	17.0						0	7.8	34.6	2
F2	1	15.17	-9.83	23.0	8.2						0	7.4	49.0	3
F3	1	25.33	0.33	26.0	24.0						3	1.2	4.6	3
F3	10	30.23	5.23	44.2	18.6	8.53					0	7.1	23.6	10

The overall mean for method B is 27.8 ± 4.8 .

Table 7A Comparison of results for sample 11 (KHP COD standard, 125 mg/l)

Method code	Laboratory number	Day 1	Day 2	Day 3	Day 4	Day 5
B _{C-T}	1	112.4	109	107.9	108.5	109.1
B _{C-T}	4	115.9	124	113.6	116.1	135.3
B _{C-T}	5	124.9	127	147.8	124	137.8
B _{C-T}	6	125	128	126	128	127
B _{C-T}	7	130	129	133	137	130
B _{C-T}	8	114.3	128.6	120.9	111.1	130.8
B _{O-T}	7	126	123	123	119	120
B _{O-T} *	7			116	117.6	116.9
D	1	121		120	112	
D	2	124	122	123	124	130
D	5	113.7		118.6		
D	6	125	121	117	116	
A	11	127.5	131.3	127.9	122.0	124.9
G	3	< 5	< 5			
F1	7	134	128	126	123	124
F1*	7	132	134	126	123	126
F1	9	130	128	120	128	126
F1	12	123	120			
F2	1	122		115		119
F3	1	95	93	101		115
F3	10	132.6	122.6	101.2	103.8	145.8
					132.6	135.2
					132.6	154.4
						141.8

Table 7B Statistical analysis of results for sample 11 (KHP COD standard, 125 mg/l)

Method code	Laboratory number	Mean	Bias	Max	Min	Sw	Sb	St	RSD %	DoF	Number of outlier results	Est SD	Est RSD %	Number of results used
B _{C-T}	1	108.08	-16.92	120.1	98.1	5.23	2.78	5.92	5.48	8	0			10
B _{C-T}	4	122.37	-2.63	142.2	113.6	3.90	8.97	9.78	7.99	5	0			10
B _{C-T}	5	134.16	9.16	147.8	124.0	8.12					0	7.6	5.7	10
B _{C-T}	6	124.00	-1.00	128.0	114.0	4.15					0	5.0	4.1	9
B _{C-T}	7	131.33	6.33	137.0	129.0	1.73	2.93	3.40	2.59	3	0			6
B _{C-T}	8	123.19	-1.81	131.4	111.1	6.74					0	6.6	5.4	10
B _{O-T}	7	122.00	-3.00	126.0	119.0	1.73	1.17	2.09	1.71	8	0			10
B _{O-T} *	7	113.83	-11.17	117.6	99.6	6.61	2.62	7.11	7.14	4	0			6
D	1	117.67	-7.33	121.0	112.0						0	4.9	4.2	3
D	2	124.38	-0.63	130.0	119.0	1.97	3.23	3.79	3.04	4	0			8
D	5	116.15	-8.85	118.6	113.7						0	3.5	3.0	2
D	6	119.75	-5.25	125.0	116.0	2.06	4.36	4.82	4.03	1	0			4
A	11	125.86	0.86	131.3	120.4	2.84	2.72	3.93	3.12	7	0			10
G	3	< 5												
F1	7	127.40	2.40	135.0	120.0	2.79	4.10	4.96	3.89	5	0			10
F1*	7	127.10	2.10	134.0	122.0	2.21	3.29	3.97	3.12	5	0			10
F1	9	127.50	2.50	135.0	120.0	3.56	2.40	4.30	3.37	8	0			10
F1	12	121.50	-3.50	123.0	120.0						0	2.1	1.7	2
F2	1	118.67	-6.33	122.0	115.0						0	3.5	3.0	3
F3	1	104.33	-20.67	125.0	92.0						0	12.8	12.3	6
F3	10	130.26	5.26	154.4	101.2	6.68	16.58	17.88	13.72	5	0			10

The overall mean for method B is 123.6 ± 8.3 .

Table 8A Comparison of results for sample 7 (KHP COD standard, 160 mg/l)

Method code	Laboratory number	Day 1	Day 2	Day 3	Day 4	Day 5
B _{C-T}	1	148.4	134.7	135.6	136	147.6
B _{C-T}	4	154.4	157.6	143.3	147	144.1
B _{C-T}	5	173	156.2	168.5	164.7	173.8
B _{C-T}	6	154	160		160	164
B _{C-T}	7	155	153	163	161	169
B _{C-T}	8	150.2	152.9	149.3	143.7	162.3
B _{O-T}	7	160	157	158	156	154
B _{O-T} *	7	159.4	151.1	149.5	149.2	156.6
D	1	154		156		154
D	2	161	162	161	162	
D	5	152.3		173.4		
D	6	162	149	152	149	
A	11	160.6	165.9#	155.9	155.6	159.7
G	3	80	130			
F1	7	164	161	163	158	160
F1*	7	165	165	165	164	162
F1	9	164		157	162	163
F1	12	154	157			
F2	1	162		163	147	
F3	1	192	188	117#		182
F3	10	163	178.6	122.6	125.2	177
					180.2	178.6
						184.8
						184.8
						187.6

Table 8B Statistical analysis of results for sample 7 (KHP COD standard, 160 mg/l)

Method code	Laboratory number	Mean	Bias	Max	Min	Sw	Sb	St	RSD %	DoF	Number of outlier results	Est SD	Est RSD %	Number of results used
B _{C-T}	1	141.66	-18.34	150.8	134.7	6.86					1	6.5	4.6	9
B _{C-T}	4	150.19	-9.81	157.6	143.3	4.54	2.20	5.04	3.36	8	0			10
B _{C-T}	5	169.27	9.27	176.7	156.2	5.61	1.48	5.80	3.43	9	0			10
B _{C-T}	6	158.75	-1.25	173.0	135.0	5.49	3.27	6.39	4.03	4	1			8
B _{C-T}	7	161.00	1.00	169.0	153.0	2.00	6.40	6.71	4.17	3	0			6
B _{C-T}	8	153.84	-6.16	162.6	143.7	2.99	5.73	6.46	4.20	5	0			10
B _{O-T}	7	156.60	-3.40	160.0	153.0	2.32	0.85	2.47	1.58	9	0			10
B _{O-T} *	7	153.74	-6.26	161.7	149.2	3.53	3.58	5.03	3.27	5	0			8
D	1	154.67	-5.33	156.0	154.0						0	1.2	0.7	3
D	2	162.25	2.25	166.0	159.0	2.12					0	2.1	1.3	8
D	5	162.85	2.85	173.4	152.3						0	14.9	9.2	2
D	6	153.00	-7.00	162.0	149.0	6.67					0	6.2	4.0	4
A	11	157.54	-2.46	160.6	154.7	0.96	1.52	1.80	1.14	4	1			9
G	3	105.00	-55.00	130.0	80.0						0	35.4	33.7	2
F1	7	161.20	1.20	165.0	157.0	2.83					0	2.7	1.6	10
F1*	7	162.70	2.70	165.0	159.0	1.76	1.42	2.26	1.39	7	0			10
F1	9	162.25	2.25	165.0	157.0	2.74					0	2.7	1.6	8
F1	12	155.50	-4.50	157.0	154.0						0	2.1	1.4	2
F2	1	154.00	-6.00	162.0	147.0						0	7.6	4.9	3
F3	1	188.00	28.00	192.0	182.0						1	4.3	2.3	4
F3	10	168.24	8.24	187.6	122.6	5.54	25.11	25.71	15.28	4	0			10

The overall mean for method B is 155.8 ± 8.7.

Table 9A Comparison of results for sample 5 (sodium acetate COD standard, 128 mg/l)

Method code	Laboratory number	Day 1	Day 2	Day 3	Day 4	Day 5
B _{C-T}	1	93.9	84.8	70	77.6	100.5
B _{C-T}	4	80	106.2	103.7	114.6	87.9
B _{C-T}	5	117.1	89.3	120.9	100.2	114.9
B _{C-T}	6	114	110		118	112
B _{C-T}	7	111	111	120	118	126
B _{C-T}	8	98.4	112.9	106.9	101.3	116.7
B _{O-T}	7	115	115	116	119	110
B _{C-T} *	7	100.9	114.3	111.3	107.3	112.8
D	1	123		114		120
D	2	123	124	126	129	122
D	5	51.3		134.9		
D	6	122	114	112	116	
A	11	92	101.6	94	91.4	102.9
G	3	130	110			
F1	7	117	110	117	113	114
F1*	7	124	124	120	121	121
F1	9	120	118	111	121	115
F1	12	96	119			
F2	1	110		110		108
F3	1	86	88	85		91
F3	10	116.4	115.2	88.4	100	131.4
						132.6
						130.2
						125.2
						135.2
						132.6

Table 9B Statistical analysis of results for sample 5 (sodium acetate COD standard, 128 mg/l)

Method code	Laboratory number	Mean	Bias	Max	Min	Sw	Sb	St	RSD %	DoF	Number of outlier results	Est SD	Est RSD %	Number of results used	
B _{C-T}	1	89.89	-38.11	108.0	70.0	8.21	7.96	11.44	12.72	7	0			10	
B _{C-T}	4	95.57	-32.43	114.6	80.0	11.65	2.65	11.95	12.50	9	0			10	
B _{C-T}	5	109.11	-18.89	127.2	82.2	15.84					0		14.5	13.3	10
B _{C-T}	6	113.88	-14.13	118.0	110.0	1.62					1	2.9	2.5	8	
B _{C-T}	7	119.17	-8.83	129.0	111.0	1.47	8.19	8.32	6.98	3	0			6	
B _{C-T}	8	112.48	-15.52	122.3	98.4	5.75	5.66	8.07	7.17	7	0			10	
B _{O-T}	7	113.20	-14.80	119.0	108.0	1.48	3.24	3.56	3.15	5	0			10	
B _{O-T} *	7	108.65	-19.35	114.3	100.9	5.01					0	4.6	4.2	8	
D	1	119.00	-9.00	123.0	114.0						0	4.6	3.9	3	
D	2	126.38	-1.63	134.0	122.0	2.21	3.65	4.26	3.37	4	0			8	
D	5	93.10	-34.90	134.9	51.3						0	59.1	63.5	2	
D	6	116.00	-12.00	122.0	112.0	4.47					0	4.3	3.7	4	
A	11	98.00	-30.00	104.6	91.4	3.63	3.25	4.87	4.97	7	0			10	
G	3	120.00	-8.00	130.0	110.0						0	14.1	11.8	2	
F1	7	114.50	-13.50	120.0	110.0	3.21					0	3.0	2.6	10	
F1*	7	118.90	-9.10	124.0	112.0	1.92	3.17	4.23	3.56	5	0			10	
F1	9	118.00	-10.00	124.0	111.0	4.17					0	3.7	3.1	10	
F1	12	107.50	-20.50	119.0	96.0						0	16.3	15.1	2	
F2	1	109.33	-18.67	110.0	108.0						0	1.2	1.1	3	
F3	1	98.33	-29.67	120.0	85.0						0	16.9	17.2	6	
F3	10	120.72	-7.28	135.2	88.4	4.11	16.15	16.67	13.81	4	0			10	

The overall mean for method B is 107.0 ± 10.7. (The negative bias from all laboratories may suggest incomplete oxidation).

Table 10A Comparison of results for sample 15 (humic acid solution)

Method code	Laboratory number	Day 1	Day 2	Day 3	Day 4	Day 5
B _{C-T}	1	94	89.2	101.2	99.6	106.6
B _{C-T}	4	105.7	106.9	101.8	101.5	112.5
B _{C-T}	5	133.5	129.9	118.7	117.9	127.9
B _{C-T}	6	112	111	101	114	107
B _{C-T}	7	106	109	124	124	125
B _{C-T}	8	110.1	100.7#	111.3	109.2	114.4
B _{O-T}	7	118	115	114	114	116.1
B _{O-T} *	7	106.7	105.7	109.8	110.4	109.2
D	1	116		117	108	
D	2	121	121	123	121	122
D	5	136.7		130.1		
D	6	117	109	113	117	
A	11	115.1	122.9	115.9	110.6	122.9
G	3	190	170			
F1	7	123	119	118	117	114
F1*	7	118	118	117	116	118
F1	9	124	118	121	114	116
F1	12	111	124			
F2	1	112		109		113
F3	1	88	101		115	89
F3	10	117.8	117.8	119	94.8	193.2
					177	130.2
						131.4
						114.6
						140.4

Table 10B Statistical analysis of results for sample 15 (humic acid solution)

Method code	Laboratory number	Mean	Max	Min	Sw	Sb	St	RSD %	DoF	Number of outlier results	Est SD	Est RSD %	Number of results used
B _{C-T}	1	100.01	120.9	88.6	6.35	7.85	10.10	10.10	6	0			10
B _{C-T}	4	108.57	118.0	101.5	3.65	4.08	5.47	5.04	6	0			10
B _{C-T}	5	122.95	133.5	109.3	6.42	3.65	7.39	6.01	8	0			10
B _{C-T}	6	105.50	119.0	87.0	11.84					0	10.5	9.9	10
B _{C-T}	7	118.17	125.0	106.0	2.04	9.14	9.36	7.92	2	0			6
B _{C-T}	8	112.90	116.8	109.2	2.82	1.57	3.22	2.86	6	1			9
B _{O-T}	7	114.00	118.0	111.0	1.95	0.69	2.07	1.81	9	0			10
B _{O-T} *	7	109.13	111.6	105.7	1.08	1.81	2.10	1.93	4	0			8
D	1	113.67	117.0	108.0						0	4.9	4.3	3
D	2	120.25	123.0	117.0	1.73	1.19	2.10	1.75	6	0			8
D	5	133.40	136.7	130.1						0	4.7	3.5	2
D	6	114.00	117.0	109.0	4.47					0	3.8	3.4	4
A	11	117.78	122.9	110.6	3.36	1.86	3.85	3.27	8	0			10
G	3	180.00	190.0	170.0						0	14.1	7.9	2
F1	7	118.70	123.0	114.0	1.64	2.82	3.27	2.75	5	0			10
F1*	7	118.20	122.0	115.0	1.34	1.64	2.12	1.79	6	0			10
F1	9	119.90	125.0	114.0	3.45					0	3.4	2.9	10
F1	12	117.50	124.0	111.0						0	9.2	7.8	2
F2	1	111.33	113.0	109.0						0	2.1	1.9	3
F3	1	102.60	120.0	88.0						0	14.6	14.3	5
F3	10	133.62	193.2	94.6	12.31	28.97	31.48	23.56	5	0			10

The overall mean for method B is 111.7 ± 7.8 .

Table 11A Comparison of results for sample 13 (chloride concentration 500 mg/l)

Method code	Laboratory number	Day 1	Day 2	Day 3	Day 4	Day 5
B _{C-T}	1	-7.3	-8.9	0.6	-4.9	-4.5
B _{C-T}	4	-3.4	-3	-0.5	1.3	0.5
B _{C-T}	5	19.6	12.5	15.7	8.1	17.4
B _{C-T}	6	16	-2	4	5	1
B _{C-T}	7	0.3	0.3	4	0.4	2.5
B _{C-T}	8	10.2	-2.5	7.2	-2.8	7.7
B _{O-T}	7	4	2	4	6	4
B _{O-T} *	7	6.1	0.3	3.7	5.6	11.4
D	1	15		16	0.26	
D	2	13	23	5	13	57
D	5	21		18.2		
D	6	8	5	3	3	
A	11	4.2	1.2	0	4.3	0
G	3	130	160			
F1	7	4	1	3	1	0
F1*	7	7	8	0	0	7
F1	9	4	2	6	2	6
F1	12	< 5	13			
F2	1	1.3		8.2		11
F3	1	< 5	< 5	< 5	< 5	< 5
F3	10	< 10	< 10	< 10	< 10	10.2
					< 10	< 10
					14.1	16

Table 11B Statistical analysis of results for sample 13 (chloride concentration 500 mg/l)

Method code	Laboratory number	Mean	Max	Min	Sw	Sb	St	RSD %	DoF	Number of outlier results	Est SD	Number of results used
B _{C-T}	1	-3.04	15.7	-18.3	4.37	9.58	10.53	-346.30	5	0		10
B _{C-T}	4	1.54	10.4	-3.4	4.48	2.17	4.98	322.65	6	0		9
B _{C-T}	5	12.44	19.6	6.5	5.69					0	4.7	10
B _{C-T}	6	13.56	37.0	-2.0	7.79	12.87	15.05	111.01	4	0		9
B _{C-T}	7	1.75	4.0	0.3	0.95	1.48	1.75	100.26	3	0		6
B _{C-T}	8	7.27	15.1	-2.8	5.53	2.98	6.28	86.38	8	0		10
B _{O-T}	7	4.90	8.0	2.0	1.14	1.91	2.22	45.41	5	0		10
B _{O-T} *	7	5.01	11.4	0.2	2.89	2.62	3.90	77.86	5	0		8
D	1	10.42	16.0	0.3						0	8.8	3
D	2	31.88	57.0	5.0	5.53	21.25	21.96	68.90	3	0		8
D	5	19.60	21.0	18.2						0	2.0	2
D	6	4.75	8.0	3.0	1.50	2.24	2.69	56.69	1	0		4
A	11	2.79	4.9	0.0	1.68	1.48	2.24	80.30	7	0		10
G	3	145.00	160.0	130.0						0	21.2	2
F1	7	2.60	7.0	0.0	1.95	1.23	2.31	88.75	8	0		10
F1*	7	3.80	8.0	0.0	2.28	2.26	3.21	84.46	7	0		10
F1	9	5.00	12.0	1.0	2.14	3.33	3.96	79.18	5	0		10
F1	12	13.00	13.0	< 5								1
F2	1	6.83	11.0	1.0						0	5.0	3
F3	1	< 5	< 5	< 5								
F3	10	13.43	16.0	< 10						0	3.0	3

The overall mean for method B is 5.5 ± 6.1 .

Table 12A Comparison of results for sample 8 (KHP COD standard 125 mg/l, chloride concentration 500mg/l)

Method code	Laboratory number	Day 1	Day 2	Day 3	Day 4	Day 5
B _{C-T}	1	109.1	109.2	106.9	109.2	108
B _{C-T}	4	118.5	113.6	112.6	113.8	116.6
B _{C-T}	5	131.3	132.7	137.1	145.9	138.9
B _{C-T}	6	130	126	120	128	133
B _{C-T}	7	124	117	137	132	123
B _{C-T}	8	128.5	121.9	111.1#	120.9	124.5
B _{O-T}	7	137#	125	124	121	121
B _{O-T} *	7	121.4	118.2	113.8	118.2	123.6
D	1	132		136	115	
D	2	139	171	129	140	166
D	5	126.4		126.7		
D	6		143	119	124	
A	11	130.7	120.7	123.9	124	122.9
G	3	210	170			
F1	7	133	121	126	124	122
F1*	7	136	133	131	130	
F1	9	128	132	122	130	149#
F1	12	127	131			
F2	1	121		123		119
F3	1	98	102	97		115
F3	10	127.6	140.4	128.8	105.2#	144.6
					147.2	141.8
						139.2
						152.8
						151.4

Table 12B Statistical analysis of results for sample 8 (KHP COD standard 125 mg/l, chloride concentration 500 mg/l)

Method code	Laboratory number	Mean	Bias	Max	Min	Sw	Sb	St	RSD %	DoF	Number of outlier results	Est SD	Est RSD %	Number of results used
B _{C-T}	1	109.69	-15.31	114.0	106.0	1.47	2.24	2.67	2.44	5	1			9
B _{C-T}	4	115.64	-9.36	118.5	112.6	1.62	0.84	1.82	1.58	8	0			10
B _{C-T}	5	138.48	13.48	147.6	131.3	4.15	3.18	5.23	3.78	7	0			10
B _{C-T}	6	126.50	1.50	133.0	117.0	4.24	4.24	6.00	4.74	5	1			8
B _{C-T}	7	126.67	1.67	137.0	117.0						0	7.1	5.6	6
B _{C-T}	8	123.88	-1.12	128.5	120.3	3.46					1	2.8	2.3	9
B _{O-T}	7	122.67	-2.33	125.0	121.0	1.06	1.73	2.03	1.66	4	1			9
B _{O-T} *	7	118.45	-6.55	123.6	113.8	3.64					0	3.2	2.7	8
D	1	127.67	2.67	136.0	115.0						0	11.2	8.7	3
D	2	155.13	30.13	189.0	129.0	15.66	13.67	20.79	13.40	5	0			8
D	5	126.55	1.55	126.7	126.4						0	0.2	0.2	2
D	6	128.67	3.67	143.0	119.0						0	12.7	9.8	3
A	11	124.20	-0.80	130.7	120.7	3.60					0	3.0	2.4	10
G	3	190.00	65.00	210.0	170.0						0	28.3	14.9	2
F1	7	126.30	1.30	133.0	121.0	4.06					0	3.7	3.0	10
F1*	7	129.88	4.88	136.0	122.0	3.92	2.28	4.54	3.49	2	0			8
F1	9	129.22	4.22	134.0	122.0	4.03					1	3.6	2.8	9
F1	12	129.00	4.00	131.0	127.0						0	2.8	2.2	2
F2	1	121.00	-4.00	123.0	119.0						0	2.0	1.7	3
F3	1	106.00	-19.00	130.0	94.0						0	13.9	13.1	6
F3	10	141.53	16.53	152.8	127.6	4.73	6.95	8.41	5.94	4	1			9

The overall mean for methods A and B is 123.4 ± 9.1 .

Table 13A Comparison of results for sample 3 (chloride concentration 2000 mg/l)

Method code	Laboratory number	Day 1	Day 2		Day 3		Day 4		Day 5		
B _{C-T}	1	3.7	-1.3	-7	-10.2	-0.3	0.2	17.2	15.7	1.9	0.2
B _{C-T}	4	-1	0.7	7.1	11.3	9.6	16.6	5.6	3.2	5.7	-2.9
B _{C-T}	5	19.6	37.3	22.7	12.6	7.9	15.2	17.8	25.7	31.1	5.4
B _{C-T}	6	14	7	13	21	12	20	2	44#	12	11
B _{C-T}	7	11	12	12	10	10	9				
B _{C-T}	8	2.9	6.4	10.3	8.2	12.3	7.5	24	20.8	8.5	7.7
B _{O-T}	7	12	10	10	11	6	9	8	10	11	7
B _{O-T} *	7	14.7	16.6	16.5	16.8	4.6	6.6	11.5	9.3		
D	1	18		7		12					
D	2	48	54	22	24	36	110#	50	53		
D	5	21.8		10.4							
D	6		5	21							
A	11	2.3	2.3	8	4.3	8.4	8	4.4	7.6	3.8	7.6
G	3	150	180								
F1	7	12	8	8	9	5	2	12	6	11	7
F1*	7	19	2	20	10	14	14	9	7	13	8
F1	9	25	27	0	0	17	34	27	26	55	60
F1	12	< 5	< 38								
F2	1	6.9		44.5		2					
F3	1	< 5	< 5	< 5		14		< 5		22	
F3	10	14.1	12.8	< 10	11.5	19.9	19.9	10.2	16	23.7	19.9

Table 13B Statistical analysis of results for sample 3 (chloride concentration 2000 mg/l)

Method code	Laboratory number	Mean	Max	Min	Sw	Sb	St	RSD %	DoF	Number of outlier results	Est SD	Number of results used
B _{C-T}	1	2.01	17.2	-10.2	2.02	8.93	9.15	455.32	4	0		10
B _{C-T}	4	5.59	16.6	-2.9	3.86	4.78	6.15	109.99	6	0		10
B _{C-T}	5	19.53	37.3	5.4	10.92					0	10.0	10
B _{C-T}	6	12.44	21.0	2.0	4.72					1	5.9	9
B _{C-T}	7	10.67	12.0	9.0	1.00	0.76	1.26	11.80	6	0		6
B _{C-T}	8	10.86	24.0	2.9	2.25	6.57	6.95	63.96	4	0		10
B _{O-T}	7	9.40	12.0	6.0	1.84	0.47	1.90	20.25	9	0		10
B _{O-T} *	7	12.08	16.8	4.6	1.25	5.04	5.19	42.98	3	0		8
D	1	12.33	18.0	7.0						0	5.5	3
D	2	41.00	54.0	22.0	2.86	16.19	16.44	40.09	3	1		7
D	5	16.10	21.8	10.4						0	8.1	2
D	6	11.00	21.0	5.0						0	8.7	3
A	11	5.67	8.4	2.3	1.96	1.61	2.54	44.80	7	0		10
G	3	165.00	180.0	150.0						0	21.2	2
F1	7	8.00	12.0	2.0	2.79	1.65	3.24	40.55	8	0		10
F1*	7	11.60	20.0	2.0	6.47					0	5.5	10
F1	9	27.10	60.0	0.0	5.65	19.99	20.78	76.66	4	0		10
F1	12											
F2	1	17.80	44.5	2.0						0	23.3	3
F3	1	18.00	22.0	< 5						0	4.0	2
F3	10	15.30	23.7	< 10	3.03	5.07	5.91	38.64	5	0	5.3	9

The overall mean for method B is 10.1 ± 5.5 .

Table 14A Comparison of results for sample 2 (final sewage effluent1)

Method code	Laboratory number	Day 1	Day 2	Day 3	Day 4	Day 5
B _{C-T}	1	38.1	40.6	41.3	38.7	47.2
B _{C-T}	4	61.6	48	42.4	40.8	54.5
B _{C-T}	5	51.6	74.4	56.4	67.9	56.7
B _{C-T}	6	49	54	53	46	50
B _{C-T}	7	57	52	55	55	54
B _{C-T}	8	40.8	53.2	57.5	76.4	60.1
B _{O-T}	7	54	54	52	54	53
B _{O-T} *	7	54.6	57.8	41.9	36.9	46.9
D	1	75		65		74
D	2	80	70	68	72	84
D	5	59.1		63.5		
D	6	77	59	67	59	
A	11	59.6	57.3	57	59.2	61.4
G	3	130	110			
F1	7	57	50	56	54	54
F1*	7	59	62	47	43	47
F1	9	52	55	59	53	61
F1	12	55	60			
F2	1	52		45.8		48
F3	1	21	19	19		59
F3	10	56.3	49.4	51.3	50	66.3
						66.3
						65.1
						58.9
						67.6
						67.6

Table 14B Statistical analysis of results for sample 2 (final sewage effluent 1)

Method code	Laboratory number	Mean	Max	Min	Sw	Sb	St	RSD %	DoF	Number of outlier results	Est SD	Est RSD (%)	Number of results used
B _{C-T}	1	43.22	49.7	38.1	3.38	2.46	4.18	9.67	7	0			10
B _{C-T}	4	52.70	62.5	40.8	6.44	4.42	7.81	14.82	8	0			10
B _{C-T}	5	60.85	74.4	51.6	8.84					0		6.8	11.2
B _{C-T}	6	54.90	76.0	44.0	9.27	4.03	10.11	18.41	8	0		1.9	3.5
B _{C-T}	7	55.00	57.0	52.0	2.38					0			6
B _{C-T}	8	58.06	76.4	40.8	7.49	5.17	9.10	15.68	8	0			10
B _{O-T}	7	53.90	56.0	52.0	0.71	1.14	1.34	2.49	5	0			10
B _{O-T} *	7	46.75	57.8	36.9	3.12	6.68	7.37	15.77	4	0			8
D	1	71.33	75.0	65.0						0	5.5	7.7	3
D	2	78.00	85.0	68.0	3.97	6.05	7.24	9.28	4	0		3.1	8
D	5	61.30	63.5	59.1						0		3.1	5.1
D	6	65.50	77.0	59.0	9.85					0		8.5	13.0
A	11	58.29	61.4	55.4	1.88	0.65	1.95	3.42	9	0		14.1	10
G	3	120.00	130.0	110.0						0			2
F1	7	55.40	60.0	50.0	2.93					0		2.9	10
F1*	7	51.70	67.0	43.0	3.45	8.65	9.32	18.02	5	0			10
F1	9	57.90	64.0	52.0	2.92	3.29	4.39	7.59	6	0			10
F1	12	57.50	60.0	55.0						0		3.5	2
F2	1	48.60	52.0	45.8						0		3.1	3
F3	1	39.17	63.0	19.0						0		21.6	6
F3	10	59.88	67.6	49.4	2.96	7.46	8.02	13.40	5	0			10

The overall mean for method B is 54.1 ± 5.5 .

Table 15A Comparison of results for sample 4 (final sewage effluent 2)

Method code	Laboratory number	Day 1	Day 2	Day 3	Day 4	Day 5
B _{C-T}	1	54.4	55.5	54	57.6	58.4
B _{C-T}	4	73.8	61.3	55.7	58.1	69.4
B _{C-T}	5	71.6	77.9	77.1	65.6	79.9
B _{C-T}	6	85	79	78	77	71
B _{C-T}	7	69	69	77	77	75
B _{C-T}	8	68.5	63.3	71.8	69	80.2
B _{O-T}	7	74	73	75	77	72
B _{O-T} *	7	75.7	62.3	53.1	56.9	52.9
D	1	92		90	89	
D	2	105	99	113	106	95
D	5	64.2		94		
D	6	91	79	76	77	
A	11	78.1	65.3	64	71.7	75.8
G	3	80	70			
F1	7	76	71	76	75	72
F1*	7	75	72	61	65	64
F1	9	73	67	76	69	72
F1	12	64	72			
F2	1	70		65	82	
F3	1	44	45	40	69	71
F3	10	76.4	74.3	70.2	70.9	87
					80.1	77.9
						82.3
						83.8
						86.2

Table 15B Statistical analysis of results for sample 4 (final sewage effluent 2)

Method code	Laboratory number	Mean	Max	Min	Sw	Sb	St	RSD %	DoF	Number of outlier results	Est SD	Est RSD (%)	Number of results used
B _{C-T}	1	61.71	77.3	54.0	1.82	8.64	8.83	14.32	4	0			10
B _{C-T}	4	63.82	73.8	55.7	5.39	1.82	5.69	8.92	9	0			10
B _{C-T}	5	74.21	79.9	65.6	4.81					0			10
B _{C-T}	6	84.40	104.0	71.0	8.09	9.25	12.28	14.73	6	0			10
B _{C-T}	7	74.17	78.0	69.0	1.22	4.40	4.56	6.15	3	0			6
B _{C-T}	8	73.92	81.3	63.3	2.36	5.52	6.00	8.12		0			10
B _{O-T}	7	73.00	77.0	71.0	0.77	1.75	1.92	2.63	5	0			10
B _{O-T} *	7	61.55	75.7	52.9	4.99	7.32	8.86	14.40	4	0			8
D	1	90.33	92.0	89.0						0	1.5	1.7	3
D	2	103.88	113.0	95.0	7.29					0	6.7	6.4	8
D	5	79.10	94.0	64.2						0	21.1	26.6	2
D	6	80.75	91.0	76.0	6.02	4.24	7.37	9.12	2	0			4
A	11	71.24	78.1	64.0	5.07	0.38	5.09	7.14	9	0			10
G	3	75.00	80.0	70.0						0	7.1	9.4	2
F1	7	74.70	78.0	71.0	1.87	1.68	2.52	3.37	7	0			10
F1*	7	67.10	79.0	56.0	3.15	7.58	8.21	12.23	5	0			10
F1	9	74.30	81.0	67.0	3.33	3.09	4.54	6.11	7	0			10
F1	12	68.00	72.0	64.0						0	5.7	8.3	2
F2	1	72.33	82.0	65.0						0	8.7	12.1	3
F3	1	59.17	86.0	40.0						0	18.7	31.7	6
F3	10	78.91	87.0	70.2	2.79	5.64	6.29	7.97	5	0			10

The overall mean for method B is 72.0 ± 7.3 .

Table 16A Comparison of results for sample 6 (final sewage effluent 3)

Method code	Laboratory number	Day 1	Day 2	Day 3	Day 4	Day 5
B _{C-T}	1	96.3	91.5	82	84.9	91.3
B _{C-T}	4	107.7	102.6	96.8	87.9	114.6
B _{C-T}	5	106.4	95.1	129.4	110.2	108.3
B _{C-T}	6	109	109	108	106	109
B _{C-T}	7	109	104	117	113	112
B _{C-T}	8	106.5	102.5	108.1	100.9	109.1
B _{O-T}	7	112	109	110	106	105
B _{O-T} *	7	89.4	85.6	103.2	102.3	107.3
D	1	129		127		128
D	2	123	170	128	132	153
D	5	104.1		117.4		
D	6	140	137	142	115	
A	11	119.2	127	116	114.7	112.6
G	3	160	150			
F1	7	116	113	112	111	110
F1*	7	120	125	119	115	109
F1	9	118	113	103	113	112
F1	12	105	113			
F2	1	107		109		102
F3	1	82	81	76		92
F3	10	109	126	77	77	157.2
						132.6
						120.2
						141.8
						133.8
						132.6

Table 16B Statistical analysis of results for sample 6 (final sewage effluent 3)

Method code	Laboratory number	Mean	Max	Min	Sw	Sb	St	RSD %	DoF	Number of outlier results	Est SD	Est RSD (%)	Number of results used
B _{C-T}	1	91.22	102.0	79.0	3.41	7.98	8.68	9.51	5	0			10
B _{C-T}	4	105.47	127.6	87.9	8.96	5.74	10.64	10.09	8	0			10
B _{C-T}	5	109.70	129.4	95.1	7.15	4.87	8.65	7.89	8	0			10
B _{C-T}	6	113.70	139.0	105.0	12.37					0	11.8	10.3	10
B _{C-T}	7	111.50	117.0	104.0	2.74	4.00	4.85	4.35	4	0			6
B _{C-T}	8	108.34	116.5	100.9	2.99	4.78	5.64	5.20		0			9
B _{O-T}	7	107.40	112.0	102.0	1.95	2.35	3.05	2.84	5	0			10
B _{O-T} *	7	99.21	107.3	85.6	2.48	7.97	8.35	8.42	3	0			8
D	1	128.00	129.0	127.0						0	1.0	0.8	3
D	2	144.00	170.0	123.0	18.93					0	16.8	11.7	8
D	5	110.75	117.4	104.1						0	9.4	8.5	2
D	6	133.50	142.0	115.0	13.58					0	12.5	9.4	4
A	11	118.09	127.0	112.6	3.54	2.62	4.41	3.73	7	0			10
G	3	155.00	160.0	150.0						0	7.1	4.6	2
F1	7	111.70	116.0	108.0	1.70	1.50	2.27	2.03	7	0			10
F1*	7	114.40	125.0	104.0	2.76	6.52	7.08	6.19	5	0			10
F1	9	114.40	120.0	108.0	2.37	2.92	3.76	3.28	6	0			10
F1	12	109.00	113.0	105.0						0	5.7	5.2	2
F2	1	106.00	109.0	102.0						0	3.6	3.4	3
F3	1	96.00	125.0	76.0						0	21.2	22.1	6
F3	10	120.76	157.2	77.0	11.73	24.96	27.58	22.84	5	0			10

The overall mean for method B is 106.8 ± 9.7 .

Table 17A Comparison of results for sample 10 (final sewage effluent 4)

Method code	Laboratory number	Day 1	Day 2	Day 3	Day 4	Day 5
B _{C-T}	1	58.1	52.5	52.2	55.4	51.6
B _{C-T}	4	81.1	63.9	48.4	48.4	66.4
B _{C-T}	5	67.2	68.7	72.6	74.1	71.9
B _{C-T}	6	59	72	75	69	70
B _{C-T}	7	75	75	80	70	69
B _{C-T}	8	72	68.5	72.9	71.2	70.2
B _{O-T}	7	71	71	72	71	70
B _{O-T} *	7	51.7	53.3	72.2	118.8#	51.3
D	1	81		85	74	
D	2	117	100	87	90	97
D	5	66.4		68.6		
D	6	91	84	74	78	
A	11	77.8	80.4	78	81.9	77.8
G	3	180	80			
F1	7	78	73	74	75	73
F1*	7	83	81	63	55	60
F1	9	72	77	71	69	70
F1	12	68	67			
F2	1	69		69		65
F3	1	42	40	37		67
F3	10	72.3	73.6	61.3	57	93.8
						84.6
						80.8
						84.6
						82.3
						84.6

Table 17B Statistical analysis of results for sample 10 (final sewage effluent 4)

Method code	Laboratory number	Mean	Max	Min	Sw	Sb	St	RSD %	DoF	Number of outlier results	Est SD	Est RSD (%)	Number of results used
B _{C-T}	1	58.39	77.4	47.9	6.81	5.66	8.85	15.16	7	0			10
B _{C-T}	4	63.31	81.1	48.4	5.57	8.21	9.92	15.67	5	0			10
B _{C-T}	5	72.98	78.5	67.2	3.04	2.08	3.68	5.04	8	0			10
B _{C-T}	6	71.44	78.0	59.0	5.16	2.57	5.76	8.07	6	1			9
B _{C-T}	7	74.00	80.0	69.0	4.76					0	4.0	5.4	6
B _{C-T}	8	72.49	77.7	68.5	1.25	2.83	3.09	4.27		0			10
B _{O-T}	7	70.20	72.0	65.0	2.14	0.52	2.21	3.15	9	0			10
B _{O-T} *	7	60.14	72.2	51.3	1.81	9.59	9.75	16.22	2	1			7
D	1	80.00	85.0	74.0						0	5.6	7.0	3
D	2	105.25	142.0	87.0	17.13	4.26	17.65	16.77	7	0			8
D	5	67.50	68.6	66.4						0	1.6	2.3	2
D	6	81.75	91.0	74.0	4.03	7.62	8.62	10.54	1	0			4
A	11	77.93	81.9	72.6	1.98	1.54	2.50	3.21	7	0			10
G	3	130.00	180.0	80.0						0	70.7	54.4	2
F1	7	73.70	78.0	70.0	1.87	1.60	2.46	3.34	7	0			10
F1*	7	72.20	87.0	55.0	3.87	11.14	12.08	16.73	4	0			10
F1	9	73.80	80.0	69.0	2.19	3.29	3.95	5.35	5	0			10
F1	12	67.50	68.0	67.0						0	0.7	1.0	2
F2	1	67.67	69.0	65.0						0	2.3	3.4	3
F3	1	49.67	71.0	37.0						0	15.1	30.4	6
F3	10	77.49	93.8	57.0	3.53	11.53	12.06	15.56	4	0	11.4	14.8	10

The overall mean for method B is 69.0 ± 5.9 .

Table 18A Comparison of results for sample 12 (final sewage effluent 5)

Method code	Laboratory number	Day 1	Day 2	Day 3	Day 4	Day 5
B _{C-T}	1	106.9	117.9	110.6	118.5	113.7
B _{C-T}	4	131.7	151.2	136.1	121.4	131.4
B _{C-T}	5	143.5	142	151.7	153.2	142.5
B _{C-T}	6	137	135	137	138	134
B _{C-T}	7	141	140	151	152	137
B _{C-T}	8	131.6	130.1	129.1	133.6	140.3
B _{O-T}	7	129	135	132	137	134
B _{O-T} *	7	115.9	98.7	121.4	117.3	116
D	1	153		169	148	
D	2	180	177	167	179	175
D	5	136.4		133.8		
D	6	158	151	152	157	
A	11	146	140.4	137.9	143.4	145.4
G	3	150	210			
F1	7	144	143	139	141	136
F1*	7	106	105	94	92	97
F1	9	143	143	136	141	143
F1	12	134	143			
F2	1	145		143		138
F3	1	156	144	160	152	156
F3	10	122.6	120.2	115.2	116.4	154.4
					167.6	151.4
					164.6	160.2
						164.6

Table 18B Statistical analysis of results for sample 12 (final sewage effluent 5)

Method code	Laboratory number	Mean	Max	Min	Sw	Sb	St	RSD %	DoF	Number of outlier results	Est SD	Est RSD %	Number of results used
B _{C-T}	1	118.25	178.3	78.8	13.57	25.05	28.49	24.09	5	0			10
B _{C-T}	4	132.54	151.2	121.4	9.35					0	8.7	6.6	10
B _{C-T}	5	141.68	153.2	127.5	4.03	6.57	7.70	5.44	5	0			10
B _{C-T}	6	132.75	140.0	117.0	3.39	8.62	9.26	6.98	2	0			8
B _{C-T}	7	143.67	152.0	137.0	1.73	6.71	6.93	4.83	2	0			6
B _{C-T}	8	132.45	140.3	128.2	3.52	2.64	4.40	3.32	7	0			10
B _{O-T}	7	133.60	137.0	129.0	2.79	0.88	2.93	2.19	9	0			10
B _{O-T} *	7	114.21	126.2	98.7	7.22	4.82	8.68	7.60	6	0			8
D	1	156.67	169.0	148.0						0	11.0	7.0	3
D	2	175.25	183.0	167.0	5.32	1.81	5.62	3.20	7	0			8
D	5	135.10	136.4	133.8						0	1.8	1.4	2
D	6	154.50	158.0	151.0	4.30					0	3.5	2.3	4
A	11	140.56	146.0	135.6	2.67	2.89	3.93	2.80	6	0			10
G	3	180.00	210.0	150.0						0	42.4	23.6	2
F1	7	142.00	146.0	136.0	1.18	3.31	3.51	2.47	4	0			10
F1*	7	106.20	127.0	91.0	2.65	13.61	13.87	13.06	4	0			10
F1	9	142.40	147.0	136.0	2.49	2.36	3.43	2.41	7	0			10
F1	12	138.50	143.0	134.0						0	6.4	4.6	2
F2	1	142.00	145.0	138.0						0	3.6	2.5	3
F3	1	153.67	160.0	144.0						0	5.4	3.5	6
F3	10	143.72	167.6	115.2	0.12	22.66	23.47	16.33	4	0			10

The overall mean for method B is 163.6 ± 8.2.

Table 19A Comparison of results for sample 14 (final sewage effluent 6)

Method code	Laboratory number	Day 1	Day 2	Day 3	Day 4	Day 5
B _{C-T}	1	81.6	84.4	88.5	86.8	85.4
B _{C-T}	4	96	93.2	87.3	84.1	91.3
B _{C-T}	5	106.4	95.4	129.4	119.4	105.4
B _{C-T}	6	105		96	116	99
B _{C-T}	7	95	96	109	110	108
B _{C-T}	8	97.7	95.8	99.1	95.2	105.6
B _{O-T}	7	103	105	103	101	97
B _{O-T} *	7	96.4	85.9	99.8	100.1	96.8
D	1	120		116		100
D	2	159	158	141	165	145
D	5	112.7		97.4		
D	6	130	118	117	115	
A	11	109	114.7	104	112.6	117.2
G	3	190	210			
F1	7	107	104	105	103	100
F1*	7	108	107	88	93	93
F1	9	110	108	105	102	108
F1	12	92	99			
F2	1	98		101		97
F3	1	74	110	72		110
F3	10	98.8	107.6	92.4	101.2	111.4
					131.4	122.6
					110.2	132.6
						130.2

Table 19B Statistical analysis of results for sample 14 (final sewage effluent 6)

Method code	Laboratory number	Mean	Max	Min	Sw	Sb	St	RSD %	DoF	Number of outlier results	Est SD	Est RSD %	Number of results used
B _{C-T}	1	89.15	117.1	72.0	3.40	14.17	14.57	16.34	4	0			10
B _{C-T}	4	94.81	107.3	84.1	6.00	4.39	7.44	7.85	7	0			10
B _{C-T}	5	111.23	129.4	95.4	8.09	6.72	10.52	9.46	7	0			10
B _{C-T}	6	105.78	118.0	95.0	9.41					0	8.6	8.1	9
B _{C-T}	7	104.50	110.0	95.0	0.71	7.79	7.83	7.49	2	0			6
B _{C-T}	8	99.07	105.6	93.1	4.67					0	4.1	4.2	10
B _{O-T}	7	100.90	105.0	97.0	1.14	2.43	2.68	2.66	5	0			10
B _{O-T} *	7	95.54	104.4	85.1	5.94	3.52	6.91	7.23	6	0			8
D	1	112.00	120.0	100.0						0	10.6	9.4	3
D	2	156.50	165.0	141.0	10.36					0	8.8	5.6	8
D	5	105.05	112.7	97.4						0	10.8	10.3	2
D	6	120.00	130.0	115.0	6.08	3.67	7.11	5.92	2	0			4
A	11	109.97	117.2	103.2	3.91	2.26	4.52	4.11	8	0			10
G	3	200.00	210.0	190.0						0	14.1	7.1	2
F1	7	105.60	110.0	100.0	2.41	2.19	3.25	3.08	7	0			10
F1*	7	100.50	112.0	88.0	2.43	3.61	8.95	8.90	4	0			10
F1	9	107.00	114.0	101.0	2.37	3.46	4.19	3.92	6	0			10
F1	12	95.50	99.0	92.0						0	5.0	5.2	2
F2	1	98.67	101.0	97.0						0	2.1	2.1	3
F3	1	93.00	110.0	72.0						0	18.9	20.3	6
F3	10	113.84	132.6	92.4	8.45	12.58	15.15	13.31	5	0	14.6	12.8	10

The overall mean for method B is 100.8 ± 7.3 .

Table 20A Comparison of results for sample 16 (final sewage effluent 7)

Method code	Laboratory number	Day 1	Day 2	Day 3	Day 4	Day 5
B _{C-T}	1	205.5	183	192.4	201.7	195.2
B _{C-T}	4	230.3	216.4	206.1	206.1	213.1
B _{C-T}	5	197.4	185	208	203.4	228.1
B _{C-T}	6	227	230	227	236	242.8
B _{C-T}	7	200	206	230	236	258.5
B _{C-T}	8	201.5	216.2	195.3	196.9	234
B _{O-T}	7	216	220	216	210	230
B _{O-T} *	7	233.8	266.3	200.1	197.6	217
D	1	243		259	218.1	226
D	2	246	268	254	278	217.1
D	5	263.9		236.8	212	210
D	6	262	238	232	236	206
A	11	220	221.2	215.9	233.6	212
G	3	230	200		231.4	204
F1	7	228	234	236	230	214
F1*	7	236	236	212	210	219.9
F1	9	232	234	228	222	214.2
F1	12	220	234		224	220
F2	1	224		254	226	222
F3	1	170	188	189	180	222
F3	10				251.2	260.6
					257.6	250
					238	256
					272.8	240#
						272.8

Table 20B Statistical analysis of results for sample 16 (final sewage effluent 7)

Method code	Laboratory number	Mean	Max	Min	Sw	Sb	St	RSD %	DoF	Number of outlier results	Est SD	Est RSD (%)	Number of results used
B _{C-T}	1	192.42	213.1	158.0	12.41	9.65	15.72	8.17	5	0			9
B _{C-T}	4	214.46	246.9	195.5	15.04	6.15	16.25	7.58	8	0			10
B _{C-T}	5	223.75	258.5	185.0	13.13	22.56	26.10	11.67	5	0			10
B _{C-T}	6	232.90	263.0	201.0	15.67					0	15.5	6.7	10
B _{C-T}	7	222.67	236.0	200.0	3.83	16.82	17.25	7.75	2	0			6
B _{C-T}	8	211.06	226.0	195.3	7.14	8.35	10.99	5.21	6	0			10
B _{O-T}	7	212.40	220.0	206.0	3.22	2.85	4.30	2.03	7	0			10
B _{O-T} *	7	219.85	266.3	197.6	11.66	20.17	23.30	10.60	4	0			8
D	1	243.33	259.0	228.0						0	15.5	6.4	3
D	2	262.75	294.0	236.0	14.05	14.52	20.21	7.69	5	0			8
D	5	250.35	263.9	236.8						0	19.2	7.7	2
D	6	241.75	262.0	232.0	12.09	7.94	14.47	5.98	2	0			4
A	11	221.58	233.5	214.0	6.09	3.69	7.12	3.21	8	0			10
G	3	215.00	230.0	200.0						0	21.2	9.9	2
F1	7	228.60	236.0	220.0	3.03	5.81	6.55	2.87	5	0			10
F1*	7	222.20	236.0	210.0	2.45	9.58	9.88	4.45	4	0			10
F1	9	230.8	256.0	220.0	3.10	13.09	13.45	5.83	4	0			10
F1	12	227.00	234.0	220.0						0	9.9	4.4	2
F2	1	234.67	254.0	224.0						0	16.8	7.1	3
F3	1	182.20	189.0	170.0						1	7.7	4.2	5
F3	10	258.83	272.8	238.0	8.87	11.11	14.22	5.49	4	0			6

The overall mean for method B is 215.7 ± 12.8 .

Table 21 **Limits of detection**

Method code	Laboratory number	Sample 9 (blank) (4.65 x Sw)	Sample 13 (500 mg/l chloride) (4.65 x Sw)	Sample 3 (2000 mg/l chloride) (4.65 x Sw)	Sample 1 (KHP COD standard, 25 mg/l) (4.65 x Sw)
B _{C-T}	1	15	20	9	33
B _{C-T}	4	12	21	18	21
B _{C-T}	5	15	26	51	25
B _{C-T}	6	34	36	22	52
B _{C-T}	7	4	4	5	7
B _{C-T}	8	14	26	10	17
B _{O-T}	7	2	5	6	4
B _{O-T} *	7	7	13	6	13
D	1				
D	2	10	26	13	10
D	5				
D	6	10	7		58
A	11	8	8	9	5
G	3				
F1	7	4	9	13	16
F1*	7	14	11	30	10
F1	9	9	10	26	15
F1	12				
F2	1				
F3	1				
F3	10			14	40

Table 22 Summary of the mean COD results for all methods (mg/l)

Sample number	COD (chloride)	Method B _{C-T} (6 labs)	Method B _{O-T} (1 lab)	Method D (4 labs)	Method A (1 lab)	Method G (1 lab)	Method F1 (3 labs)	Method F2 (1 lab)	Method F3 (2 labs)
13	(500)	6	5	17	3	145	7	7	13
3	(2000)	10	9	20	6	165	18	18	17
2	(80)	54	54	69	58	120	57	49	50
4	(80)	72	73	89	71	75	72	72	69
6	(209)	107	107	129	118	155	112	106	108
10	(350)	69	70	84	78	130	72	68	64
12	(106)	134	134	155	141	141	141	142	149
14	(368)	101	101	123	110	200	103	99	103
16	(440)	216	212	250	222	215	229	235	221
8	125 (500)	124	123	135	124	190	128	121	124
9	blank (<10)	3	1	0	1	< 5	2	7	7
1	25	28	25	27	26	118	24	15	28
11	125	124	122	120	126	< 5	126	119	117
5	128	107	113	114	98	120	113	109	110
7	160	156	157	158	158	105	160	154	178
15	(<10)	111	114	120	118	180	119	111	118

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