2020

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CENTRAL REVENUES CONTROL LABORATORY

Hill Side Road, IARI Campus, Pusa,
New Delhi-110012

New Analytical Techniques in Revenue Laboratories

...moving with the time

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FOREWORD

The role of Customs has undergone a paradigm shift from regulation to facilitation and intervention by exception. Commodity identification based on testing, whenever required, is an integral part of Customs functions which needs the support service of lab facilities. In a modern era of digital interface, Customs offices should house state of art testing facilities of all commodities which can produce quick and accurate results. This is also expected from us by our stakeholders.

To fulfil the said aspirations of our stakeholders and to strike a right balance between facilitation and enforcement, the Central Revenues Control Laboratory, New Delhi and eleven other Revenue Laboratories have a crucial role to play. These laboratories provide the required impetus in enforcement of the Customs Act, 1962, NDPS Act, 1985 and allied Acts.

The Customs department has taken several steps, in the recent past, to upgrade and strengthen its Revenue Laboratories. These laboratories have been equipped with new, state of art testing equipment/ facilities at a cost of about Rupees seventy crore, in line with the recommendations made in the WCO Laboratory Guide, 2017. This has enabled the laboratories to adopt new analytical techniques and improved procedures. The positive impact of these efforts is already visible - in the past one year itself, eight of the Revenue Laboratories have been accredited by the NABL as per ISO/IEC 17025:2017 for defined scope of tests.

This publication showcases the new facilities and equipment, which are, now, available with the Revenue Laboratories. The advantages and improvements accruing out of the use of new techniques and equipment have been illustrated with the help of real cases. This would increase awareness and the field formations would be better informed about the new and improved capabilities of the Revenue Laboratories.

I commend the efforts of CRCL and all officers involved in designing and publishing this useful manual. I also urge the field formations to fully utilize the new facilities that the Revenue Laboratories offer to ensure greater compliance and expeditious clearances.

M. Ajit Kumar
Chairman
Central Board of Indirect Taxes & Customs

CONTENTS

Scanning Electron Microscope with EDAX (SEM-EDAX)

01

Year of Purchase and Installation
2019

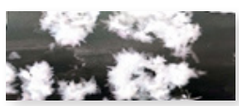
Model/OEM
EVO 18/ Carl Zeiss, U.K

Location of Instrument
CRCL New Delhi

Field of major application
Minerals, Textiles, Metals, Coatings, Polymers

The scanning electron microscope (SEM–EDAX) is one of the most versatile instruments available for the examination and analysis of the microstructure & morphology. Analysis of chemical composition & characterization is also possible with EDAX.

Practical Application

Sr. No	Description with details	Reason for analysis	Procedure adopted before instrumentation	Procedure after installation of instrument
01	Natural Calcite Powder/ Limestone	To differentiate between natural Calcite Powder/ Limestone from precipitated Calcium Carbonate of CTH 28365000 as both products are having different duty structure	The samples were analyzed for chemical composition and oil absorption On the basis of chemical composition and oil absorption, differentiation between the two was not possible	SEM imaging can differentiate between the two.  Precipitated Calcium carbonate  Natural calcite powder
02	Umbrella cloth/water proofing fabric	To differentiate between woven cloth of polyester and coated/ surface treated fabric	Ordinary microscope. However, Laboratory was unable to get high magnification images by these ordinary microscopes, hence positive identification was not possible	By SEM, 30 kx images were produced and studied for microstructure for coating and surface permeability, thereby positively identifying as coated/ water proof fabric 

CRCL Brochure_27thMay 2020_v11 jk-BNP.indd 5 6/17/2020 5:09:00 PM

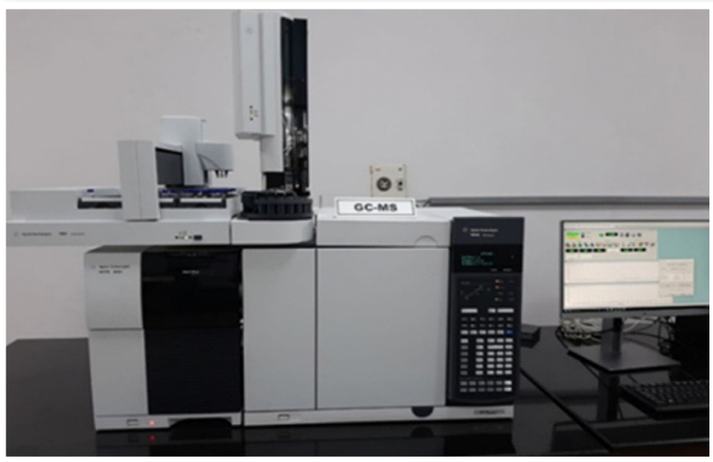
02 Gas Chromatography –Mass Spectrometer (GCMS)

Year of Purchase and Installation
2018

Model/OEM
5977B MSD Agilent technologies

Location of Instrument
CRCL New Delhi, CHL Kolkata, Mumbai, Chennai, Kochi, Vadodara, Vizag

Field of major application
Organic compounds, Food residues like Pesticides, Antibiotics, NDPS



GC-MS is the analytical technique of choice for smaller and volatile molecules. It is mainly used to identify and quantify narcotics, drugs, psychotropic substances, pesticides, pharmaceuticals, hazardous chemicals, etc.

Practical Application

Sr. No	Description with details	Reason for analysis	Procedure adopted before instrumentation	Procedure after installation of instrument
01	Sample described as Methylene Chloride	To detect Pesticide (Insecticide/ Herbicides) in the sample	Basic physical parameters were used to identify such samples i.e. Boiling Point/Melting Point, functional group test etc. Due to this limitation, exact identification was not possible	The sample was analyzed by using GC-MS Technique and FTIR and found to be 2, 4- dichlorophenoxy acetic acid (2,4 D) - a systematic herbicide, which is covered under the Insecticide Act 1968
02	Unknown samples received from UNODC, Vienna under International Collaborative Exercise (ICE)	To identify and quantify available narcotic drugs/psychotropic substances in sample	Due to lack of adequate instrumentation, CRCL was not participating in ICE. After availability of this instrument, CRCL has started participating in this programme	By using GC-MS, these unknown samples were identified, without using any Certified Reference Material . Various drugs like Cocaine, MDPV, Methamphetamine and Amphetamine have been quantified and excellent 'Z' score is secured.
03	Essential oils like Lemon grass oils, Rosemary oil, Lavender oil, Juniper berry oil and other natural essential oils	Characterization and identification of essential oil for the purpose of classification & to differentiate between products of 3301 & 3302	Only physical parameters like boiling point, refractive index, specific gravity were used to analyze and samples were reported as odoriferous substances.	By application of GC-MS, all essential oils are specifically identified and their purity is determined.

CRCL Brochure_27thMay 2020_v11 jk-BNP.indd 6 6/17/2020 5:09:02 PM

X-Ray Diffractometer (XRD)

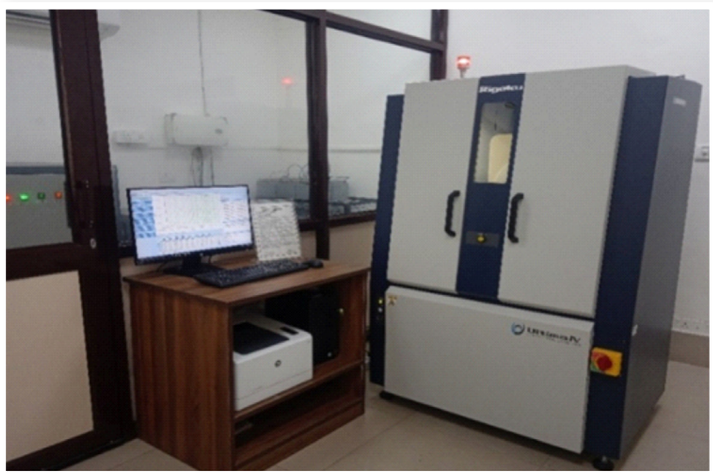
03

Year of Purchase and Installation
2018

Model/OEM
Ultima IV/Rigaku

Location of Instrument
CRCL New Delhi

Field of major application
Minerals, Ores/ Concentrates/slags



X-ray Diffractometer (XRD) is used to determine the atomic and molecular structure of a crystal/ powder, in which the crystalline structure causes a beam of incident X-rays to diffract into specific directions. By measuring the angles and intensity of these diffracted beams, identification of the minerals is carried out.

Practical Application

Sr. No	Description with details	Reason for analysis	Procedure adopted before instrumentation	Procedure after installation of instrument
01	Natural abrasive	To ascertain, whether the sample is natural abrasive of 25132090 or Garnet of 25132030 (Since Garnet is restricted for Export)	Basic chemical composition like iron, silica, and alumina used to be analyzed by wet chemistry methods. However, crystallographic study was not possible, hence exact mineralogical species could not be identified	The sample was analyzed by using XRD and identified as Garnet – a mineral, which is restricted for exports.
02	Iron ore	To ascertain iron ore with mineralogical studies	Only wet chemistry methods were applied to quantify Iron content, moisture and other physical parameters	Crystallographic studies were done by XRD and reported as Hematite
03	Sample of natural Gypsum	To ascertain, whether it is pure gypsum or added with other Minerals	Only wet chemistry methods were applied to quantify Calcium Sulphate content, moisture and other physical parameters	Crystallographic studies were done by XRD and presence of other minerals was ruled out

CRCL Brochure_27thMay 2020_v11 jk-BNP.indd 7 6/17/2020 5:09:02 PM

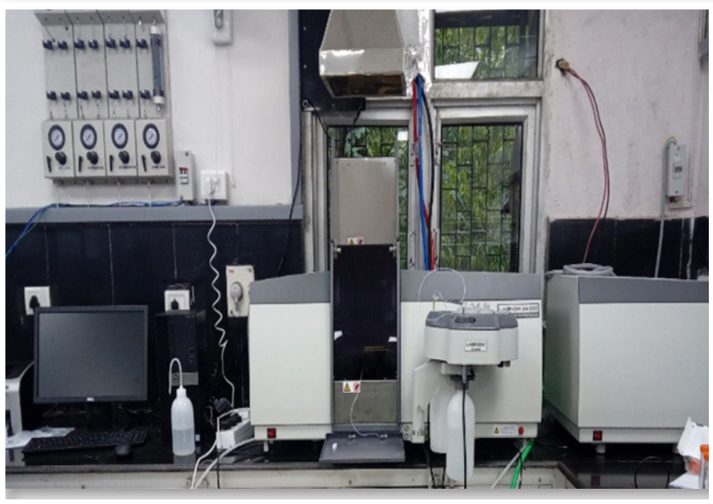
04 Atomic Absorption Spectrophotometer (AAS)

Year of Purchase and Installation
2019

Model/OEM
Lab India

Location of Instrument
CRCL New Delhi, CHL Chennai, Kochi, Goa, Kolkata, Vizag, Vadodara, Mangalore, Tuticorin

Field of major application
Inorganic materials, metals, micronutrients



Atomic Absorption Spectrometry (AAS) is an analytical technique that measures the concentrations of elements. The technique makes use of the wavelengths of light specifically absorbed by an element. Concentration of elements is measured at ppm (parts per million) levels.

Practical Application

Sr. No	Description with details	Reason for analysis	Procedure adopted before instrumentation	Procedure after installation of instrument
01	Samples of Gold-plated idols, in frame 	To analyze % Gold content along with other elements and complete composition of sample. (This was required to ascertain Gold content for purpose of valuation)	Only qualitative analysis of this type of sample for its composition i.e. Polymer substrate and Metallic (Gold) coating used to be analyzed by wet chemistry methods	The sample was analyzed by using AAS and FTIR and reported as:Idols of God, composed of Polycarbonate and it is Plated with Gold, having Gold content in the range of 0.1 % to 0.7%by wt.) (analysis of such a small quantity is possible with this instrument)
02	Sample of waste/ scrap of metallic sheets	To ascertain, whether the sample is steel sheet or Silicon Electrical steel (CRGO). The purpose of department was to ascertain, whether the material imported is CRGO (Silicon electrical steel) sheet in the guise of steel sheet/scrap	Only wet chemistry methods were used to analyze chemical composition of these type of samples. To determine silica at lower level was not possible, without instrumentation	The sample was analyzed by using AAS and reported as Silicon electrical steel

CRCL Brochure_27thMay 2020_v11 jk-BNP.indd 8 6/17/2020 5:09:03 PM

Thermogravimetric Analyzer (TGA)

05

Year of Purchase and Installation
2019

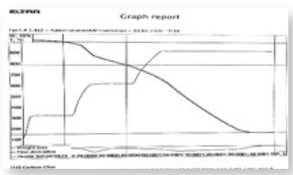
Model/OEM
Thermostep/Eltra

Location of Instrument
CRCL New Delhi, CHL Chennai, Goa

Field of major application
Polymer/ Coal / minerals/ any Thermally Decomposable matter

Thermogravimetry (TGA) is a technique that measures the change in weight of a sample as it is heated, cooled or held at constant temperature. Its main use is to characterize materials with regard to their composition. Application areas include plastics, elastomers and thermosets, mineral compounds etc.

Practical Application

Sr. No	Description with details	Reason for analysis	Procedure adopted before instrumentation	Procedure after installation of instrument
01	Samples declared as activated carbon and carbon Char	To ascertain, whether the sample is activated carbon/ carbon char as declared by the importer. Complete composition of the sample was also required to be ascertained to confirm, whether the sample is hazardous chemical	Only identification and activation test could be carried out for such samples Quantification of carbon content and other additives/residue was not possible by wet chemistry methods	The sample was analyzed by TGA and its following components were identified/ quantified 1. Volatile below 300C includes, rubber processing oil and sulphur 2. Volatile between 301C -600C includes, rubber and other polymers 3. Volatile between 601C -900C includes, Carbon black 4. Residue at 900C Silica and inorganic matter On this basis sample found to be carbon black with rubber waste, which is hazardous in nature 
02	Sample of Coal and coke	Proximate analysis (Ash, Moisture, Volatile matter)	Earlier proximate analysis was done by manual methods, and as such, results were not precise	All samples of coal, coke, were analyzed for proximate analysis and reported

CRCL Brochure_27thMay 2020_v11 jk-BNP.indd 9 6/17/2020 5:09:04 PM

06

Inductively Coupled Plasma – Mass Spectrometer (ICP-MS)

Year of Purchase and Installation
2018

Model/OEM
Agilent Technologies

Location of Instrument
CRCL New Delhi

Field of major application
Metals, food, petroleum

Inductively coupled plasma mass spectrometry (ICP-MS) atomizes the sample and creates atomic and small polyatomic ions, which are then detected by mass spectrometer. It can detect metals and several non-metals at very low concentrations i.e. PPB (Parts Per Billion). It can also detect different isotopes of the same element. It is used for simultaneous determination of elements in one test.

Practical Application

Sr. No	Description with details	Reason for analysis	Procedure adopted before instrumentation	Procedure after installation of instrument
01	Rubber Process oil	The samples which do not meet the specification of RPO (Rubber process oil) as per Indian standard IS:15078 need to undergo screening for heavy metals like Arsenic, Cadmium, Lead, Chromium in sample, to rule out , whether the sample is hazardous or not as per Environmental Protection Act.	Earlier laboratory was not able to quantify these metals in RPO samples, as instrumental analysis facility for trace level metal analysis was not available	By ICP-MS these samples are now analysed and exact quantification of these heavy metals helps to determine, whether sample is hazardous or not.
02	Samples of Turmeric powder	Heavy metals content like Arsenic , Cadmium, Lead, to ascertain toxicity for edible purpose	Unable to quantify Arsenic, Cadmium, Lead	Heavy metals like As, Cd, Pb have been quantified at the level of PPB
03	Sample of Pharmaceutical formulations / Active pharmaceutical ingredients (API)/ Ayurvedic medicines	Heavy metals content like Arsenic , Cadmium, Lead, to ascertain Toxicity, for applications as medicine	Unable to quantify toxic heavy metals like Arsenic, Cadmium, Lead	Heavy metals like As, Cd, Pb have been quantified at the PPB level

CRCL Brochure_27thMay 2020_v11 jk-BNP.indd 10 6/17/2020 5:09:05 PM

Liquid Chromatography– Mass Spectrometer – MS (LC-MS/MS)

07

Year of Purchase and Installation
2018

Model/OEM
Shimadzu

Location of Instrument
CRCL New Delhi, CHL Nhava-Sheva, Vadodara

Field of major application
Pesticides, Antibiotics, mycotoxins, NDPS

Liquid Chromatography with tandem mass spectrometry (LC-MS-MS) is a powerful analytical technique that combines the separating power of liquid chromatography with the highly sensitive and selective mass analysis capability of triple quadrupole mass spectrometry. Identification and quantification by this instrument is mandatory under FSSAI regulations in toxic and residual organic compounds in food.

Practical Application

Sr. No	Description with details	Reason for analysis	Procedure adopted before instrumentation	Procedure after installation of instrument
01	Dioctyl - erpthalate	To differentiate between Dioctylorthopthalate of CTH 29173200 and Dioctylisopthalate/ terpthalate of 29173920. (29173920 is exempted (@ 7.5%) from BCD vide Notification no 152/2009, while 29173200 is covered for exemption only @1.25%)	Basic physical parameters like boiling point, specific gravity, refractive index used to be analyzed. But these parameters cannot differentiate between these Phthalate Plasticizers, as their physical constants are very close to each other. Due to this limitation, exact identification was not possible	By using LCMS/MS sample was identified as Di octyl orhopthal-ate with purity of 99 %.
02	Hydrocodone in original Medicinal strip	To identify narcotic/ psychotropic substance in sample	Earlier these types of samples could not be identified due to non availability of LC-MS/MS	Found to be Tramadol Hydrochloride– (a Psychotropic Substance) and quantified

CRCL Brochure_27thMay 2020_v11 jk-BNP.indd 11 6/17/2020 5:09:06 PM

08 High Performance Thin Layer Chromatography Mass Spectrometer – HPTLC-MS

Year of Purchase and Installation
2018

Model/OEM
Camag/Shimadzu

Location of Instrument
CRCL New Delhi, CHL Nhava-Sheva, Vizag, Vadodara

Field of major application
Pesticides, Drugs, Pharmaceuticals, Unknown organic compounds in food, natural products

This equipment is the most advanced form of Thin Layer Chromatography. Further coupling of High Performance Thin Layer Chromatography with Mass Spectrometry provides a high degree of sensitivity and accuracy in analysis of samples.

Practical Application

Sr. No	Description with details	Reason for analysis	Procedure adopted before instrumentation	Procedure after installation of instrument
01	Sample of Ephedrine & Pseudoephedrine	Detection of Ephedrine & pseudoephedrine in sample. Ephedrine, and Pseudoephedrine are the precursors of amphetamine & methamphetamine , which are covered under Narcotic Drugs & Psychotropic Substance	Samples were tested by manual Thin Layer Chromatography (TLC), for identification as Ephedrine and Pseudoephedrine	By application of HPTLC, Ephedrine & Pseudoephedrine were seperated,detected and quantified by mass spectrometer.
02	Chilli powder for detection of illegal Dyes	Detection of Illegal dyes (Sudan I-IV)) in chilli powder, as these dyes are toxic (Carcinogenic) in nature	Laboratory was unable to detect small quantity of illegal dyes, in chilli powder, as these are added in micro level i.e. in ppm level	In samples of chilli powder detection and quantification of Sudan dyes (I-IV) was done.

CRCL Brochure_27thMay 2020_v11 jk-BNP.indd 12 6/17/2020 5:09:07 PM

Fourier Transform – Infrared Spectrometer (FT-IR)

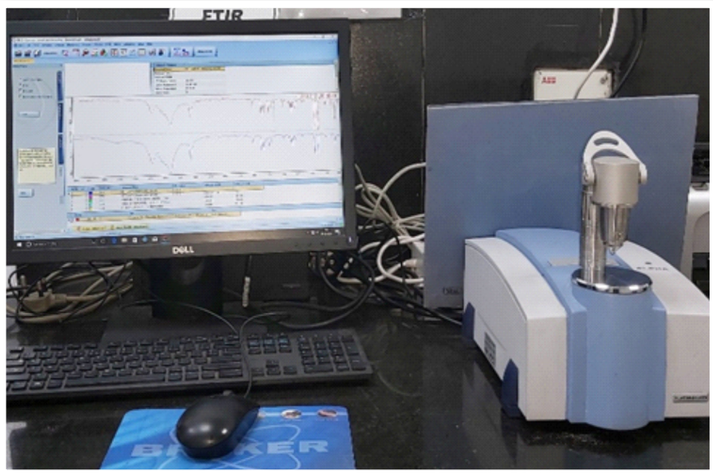
09

Year of Purchase and Installation
2018

Model/OEM
Bruker

Location of Instrument
CRCL New Delhi, CHL Kolkata, Chennai, Mumbai, Kandla, Kochi, Vizag

Field of major application
Pesticides, drugs, Pharmaceuticals, NDPS,Polymers



Identification of polymers and organic compounds in short time (01 minutes) is done by FTIR. This instrument can analyze above materials/substances in solid/liquid state.

Practical Application

Sr. No	Description with details	Reason for analysis	Procedure adopted before instrumentation	Procedure after installation of instrument
01	Samples of Bio Diesel containing Mineral hydrocarbon oil	To determine % bio-diesel content (Fatty acid methyl esters) to differentiate between Bio-Diesel of 38260000 (Mineral hydrocarbon oil content less than 70%) and 27101930 (Mineral hydrocarbon oil content more than 70%), as there is difference in structure of duties	Physical parameters used to be analyzed for such samples i.e. Boiling Point, Density, Flash Point, functional group test etc. were carried out. However, detection and quantification of Fatty acid methyl esters was not possible	By using FTIR, quantification of % Bio diesel (Fatty acid methyl esters) in the sample is done. Accordingly sample reported as mineral hydrocarbon oil without bio diesel.
02	Sample of Gold coated/plated plastic idols	To identify type of polymer substrate, on which plating is done	Physical parameters like melting point, burning characteristics, functional group (colour test) used to be analyzed, but exact identification of polymer was not possible	By using FTIR, polymer substrate is found to be composed of Polycarbonate
03	Polymer Film	To identify type of polymer, whether it is Polyethylene, Polypropylene or Polyester	Earlier, Melting Point, burning characteristics, functional group test etc. were analyzed	Identified as polyester film

CRCL Brochure_27thMay 2020_v11 jk-BNP.indd 13 6/17/2020 5:09:08 PM

10 High Performance Liquid Chromatograph (HPLC)

Year of Purchase and Installation
2018

Model/OEM
Shimadzu

Location of Instrument
CRCL New Delhi, CHL Mumbai, Kolkata, Chennai, Kochi, Vizag, Kandla, Goa, Mangalore, Tuticorin, Vadodara

Field of major application
Drugs, Pharmaceuticals, Banned Azo Dyes, Polyaromatic hydrocarbons (PAH)

High-performance liquid chromatography (HPLC) is used to separate, identify, and quantify each constituent of a mixture of organic compounds. Availability of HPLC has enabled some of the Revenue Laboratories to obtain NABL accreditation for testing of azo dyes.

Practical Application

Sr. No	Description with details	Reason for analysis	Procedure adopted before instrumentation	Procedure after installation of instrument
01	Samples of rubber process oil	The samples which do not meet the specification of Rubber Process Oil as per Indian standard IS: 15078 need to undergo screening for presence of PAH (poly aromatic hydrocarbons), as PAH above 50 PPM is considered to be hazardous (carcinogenic)	Earlier revenue laboratories were not able to test PAH in RPO samples.	By application of HPLC, sample of RPO is analysed and quantification of PAH (Poly aromatic hydrocarbon) is done. On this basis hazardous nature of sample is determined
02	Sample of textiles/ apparel, Toys	For detection and quantification of banned azo dyes. These dyes are carcinogenic in nature (total 24 types of azo dyes need to be screened)	Earlier laboratory was unable to test banned azo dyes in textile/ apparel, toys.	By application of HPLC, samples of textiles/apparels have been analysed for all 24 types of azo dyes.

CRCL Brochure_27thMay 2020_v11 jk-BNP.indd 14 6/17/2020 5:09:08 PM

Gas Chromatograph – Flame Ionization Detector / Electron Capture Detector (GC-FID/ECD)

11

Year of Purchase and Installation
2019

Model/OEM
Agilent Technologies

Location of Instrument
CRCL New Delhi, CHL Kolkata, Kandla, Mumbai, Vizag, Kochi, Chennai, Mangalore, Goa, Tuticorin

Field of major application
Pesticides, Drugs, Pharmaceuticals, Petroleum

Gas chromatography (GC) is a common type of chromatography used in analytical chemistry for separating and analyzing compounds that can be vaporized without decomposition.

Practical Application

Sr. No	Description with details	Reason for analysis	Procedure adopted before instrumentation	Procedure after installation of instrument
01	Preparation based on mineral hydro-carbon oil	To detect small quantity (1-2%) of additives like fatty acid methyl esters in the sample, so as to differentiate between mineral hydrocarbon oil and preparation based on mineral hydrocarbon oil	Basic physical parameters i.e. boiling point, density, flash point, functional group test etc. used to be analyzed. However, detection and quanti-fication of small quantity of Fatty acid methyl esters was not possi-ble in the sample.	% Fatty acid methyl esters in samples were quantified by GC-FID and found to be in the the range of 1-2%. Hence the sample is a preparation based on mineral hydrocarbon oil.
02	Neem Oil, Mahua Oil, Castor Oil	To verify nature and composition of the sample	Basic physical parameters were used to identify this type of samples i.e. refractive index, spe-cific gravity, free fatty acid content, iodine value etc. Exact identifica-tion was not possible.	Fatty acid methyl esters of each vegetable oil have been analysed by GC-FID and their fatty acid profiles are obtained On the basis of fatty acid profile each type of vegetable oil is specifi-cally confirmed with the description

CRCL Brochure_27thMay 2020_v11 jk-BNP.indd 15 6/17/2020 5:09:09 PM

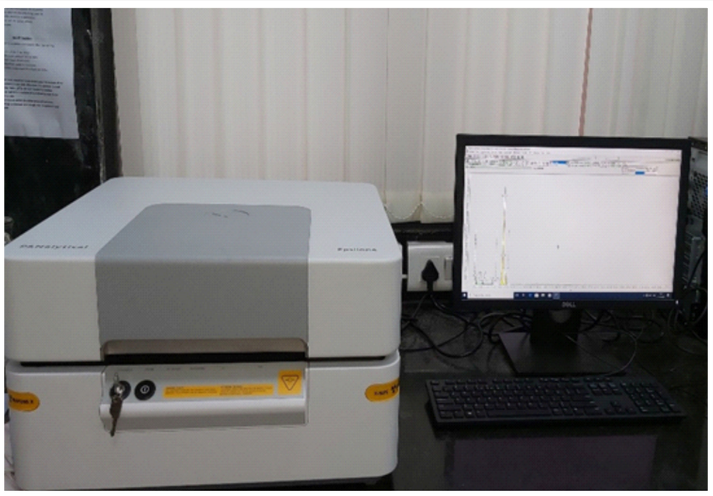
12 X-Ray Fluorescence (XRF) Spectrometer

Year of Purchase: **2019**
Year of Installation: **2020**

Model/OEM
PANalytical

Location of Instrument
CRCL New Delhi, CHL Chennai, Nhava-Sheva, Kolkata, Vadodara

Field of major application
Mineral, Ores, Metals, Sulphur in Petroleum



X-ray Fluorescence (XRF) is an analytical technique that uses the interaction of x-rays with a material to determine its elemental composition. XRF is suitable for analysis of solids, liquids and powders. It is a non-destructive analytical technique.

Practical Application

Sr. No	Description with details	Reason for analysis	Procedure adopted before instrumentation	Procedure after installation of instrument
01	Samples of refractory material in powder form	To confirm whether the sample is chrome ore of CTH 2610 or chrome ore -magnesia based refractory material of CTH 3816 (As Export duty is leviable on chrome ores)	Earlier laboratory was using wet chemistry methods. It was not possible to quantify all elements present and to differentiate between refractory materials and ores	By XRF, complete chemical composition of the sample is determined and sample is reported as refractory material
02	Samples of Petroleum products for Sulphur content	To determine Sulphur content, which is a critical environmental parameter for quality of fuel	Earlier, Sulphur content in the sample could not be determined.	By XRF, Sulphur content in PPM level is quantified in petroleum products like Automotive diesel fuel/ kerosene/ fuel oils/ light diesel oil etc.

CRCL Brochure_27thMay 2020_v11 jk-BNP.indd 16 6/17/2020 5:09:10 PM

Optical Emission Spectrometer (OES)

13

Year of Purchase: **2019**
Year of Installation: **2020**

Model/OEM
Metal power

Location of Instrument
CRCL New Delhi, CHL Kolkata

Field of major application
Metals, Alloys

Optical Emission Spectroscopy (OES), is a well trusted and widely used analytical technique used to determine the elemental composition of a broad range of metals. The type of samples which can be tested using OES include samples of tubes, bolts, rods, wires, plates and many more.

Practical Application

Sr. No	Description with details	Reason for analysis	Procedure adopted before instrumentation	Procedure after installation of instrument
01	Samples of stainless steel sheet	To determine grade of stainless steel sheet (This is required for effective implementation of Steel and Steel Products. Quality Control Order, 2020)	Earlier laboratory was not able to determine full range of metallic elements, to determine exact grade as per ASTM/ISO/BIS.	By OES, all grades of steel samples can be analysed for complete metallic composition including carbon, sulphur, phosphorous for deciding the grades of steel
02	Aluminum sheet	To quantify aluminum content with alloying metals	Earlier, quantification of alloying elements of sample was not possible.	By OES, quantification of all alloying elements is done. The sample is found to be aluminium alloy.

CRCL Brochure_27thMay 2020_v11 jk-BNP.indd 17 6/17/2020 5:09:11 PM

14

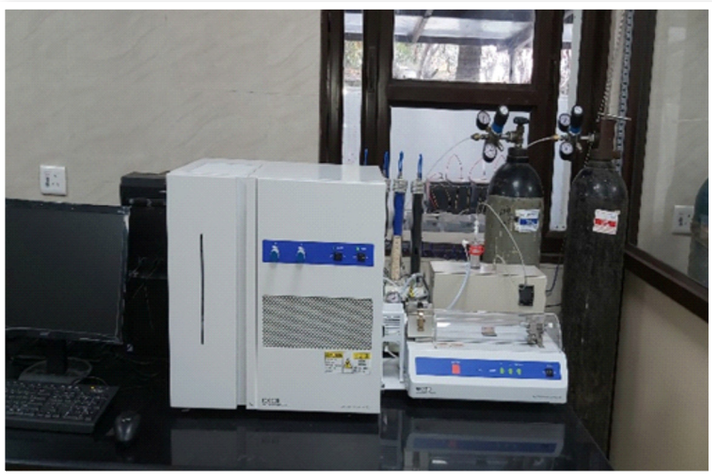
AOX Analyzer

Year of Purchase and Installation
2019

Model/OEM
Mitsubishi Chemical Analytech

Location of Instrument
CRCL New Delhi, Kolkata , Kandla

Field of major application
Waste Oil/Sludge Oils for total organic halides



AOX/TOX analyzer is used to detect all organic halides i.e. chlorine, bromine & iodine in the samples of Waste oil/Used oil, by coulometric titration, as per ASTM/EPA test methods.

Practical Application

Sr. No	Description with details	Reason for analysis	Procedure adopted before instrumentation	Procedure after installation of instrument
01	Sludge oil/waste oil	To detect and quantify total organic halides in the sample, as organic halides are hazardous (carcinogenic) in nature. (CBIC circular no. 33/ 2001-Cus. Dt. 4.06.2001)	Earlier laboratory was not able to test organic halides in this type of samples.	By AOX analyzer, samples of waste oil/sludge oils have been analyezd and organic halides have been reported at the PPM level

CRCL Brochure_27thMay 2020_v11 jk-BNP.indd 18 6/17/2020 5:09:12 PM

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CRCL Brochure_27thMay 2020_v11 jk-BNP.indd 20 6/17/2020 5:09:13 PM