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Product Information

Desktop X-ray Diffractometer "MiniFlex+"

1 . Introduction

The MiniFlex+ was developed with a totally new philosophy; it is essentially "Plug 'n Play" XRD. The MiniFlex+ is small and lightweight; it is about 1/20th the volume and 1/10th the weight of conventional X-ray diffractometers. Its design philosophy is similar to that of SONY's "Walkman", a miniaturized stereo system which allows the use of stereos in ways no one had even thought possible before. Likewise, Rigaku foresees that many new applications for XRD will be developed because of the portability and low cost of this high performance system.

Although the MiniFlex+ (Fig. 1) is new, Rigaku has analyzed a variety of samples from diverse industrial sectors demonstrating its outstanding performance as will as its features and benefits. The MiniFlex+ is an analytical system that can satisfy both the experienced XRD analyst and the beginner. The MiniFlex+ is not a replacement for Rotating Anode Systems, Microdiffractometers, or other advanced XRD systems; rather, it is a general purpose powder XRD system that can be used as the workhorse for a wide array of applications. And, because of its small size weight, and price, it will open many new doors for the use of X-ray diffraction.

Fig. 1 Desktop X-ray diffractometer "MiniFlex+".

2. Principles of X-ray Diffraction

X-ray diffraction and X-ray fluorescence (XRF) share similar principles; however, the two are used for very different applications. Figure 2 illustrates the principles of the X-ray diffraction and the X-ray fluorescence spectrometry.

When X-rays impinge upon a sample, secondary X-rays (fluorescent X-rays) which have wavelengths inherent to the sample's constituent elements will be emitted. If these fluorescent X-rays are spectrally divided with an analyzing crystal, it allows the

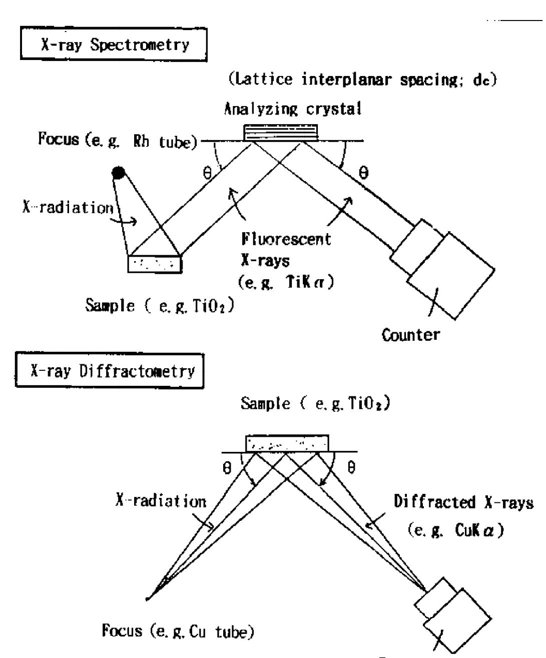


Fig. 2 Principles of X-ray spectrometry and X-ray diffractometry.

detection of diffracted X-rays at angles that satisfy the following condition of Bragg's equation.

$$2d_c \cdot \sin\theta = \lambda$$

where d_c: Lattice interplanar spacing of the analyzing crystal

θ: X-ray spectroscopic angle (Bragg angle)

λ: Wavelength of fluorescent X-rays

The fluorescent X-ray wavelength λ can be calculated by measuring the Bragg angle θ, and the sample's constituent elements can be analyzed from the wavelength λ. This is the basic principle of X-ray fluorescence spectrometry.

Suppose, on the other hand, the sample is a polycrystalline body which is an aggregate of tiny crystals and is irradiated with X-rays having a specific wavelength (e.g. CuKα rays). Then, by changing the X-ray incidence angle with respect to the sample, diffracted X-ray will be detected sequentially and those angles where each lattice plane of the crystal satisfies the Bragg condition.

$$2d \cdot \sin\theta = \lambda_0$$

Where d: Lattice interplanar spacing of crystal (sample)

θ: X-ray incidence angle (Bragg angle)

λ_0: Wavelength of characteristic X-rays due to the X-ray tube

The lattice interplanar spacing d can be calculated by measuring the Bragg angle θ. By referring the pattern of the lattice interplanar spacing d to standard data such as ICDD (International Center for Diffraction Data) data, it is possible to identify these small crystals. This is the basic principle behind X-ray diffraction.

3. Applications of X-ray Diffraction

Quartz glass (SiO_2) can be found in different forms, such as in the amorphous or crystalline state. If the amount of Silicon in the two different forms is the same, they cannot easily be distinguished from each other by the many methods of elemental analysis available. However, XRD can readily distinguish structures of materials such as these forms of SiO_2, Figs. 3 and 4 illustrate examples of this capability.

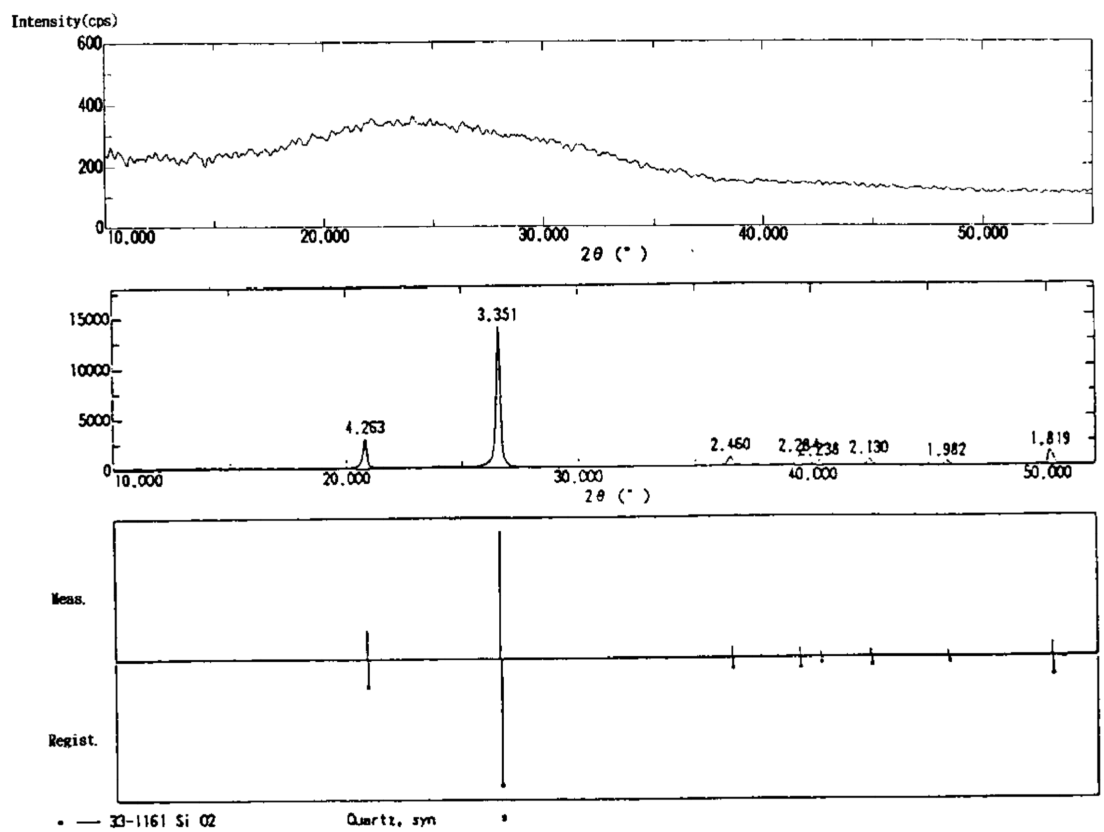


Fig. 3 difference in diffraction pattern between quartz glass and quartz (crystal).

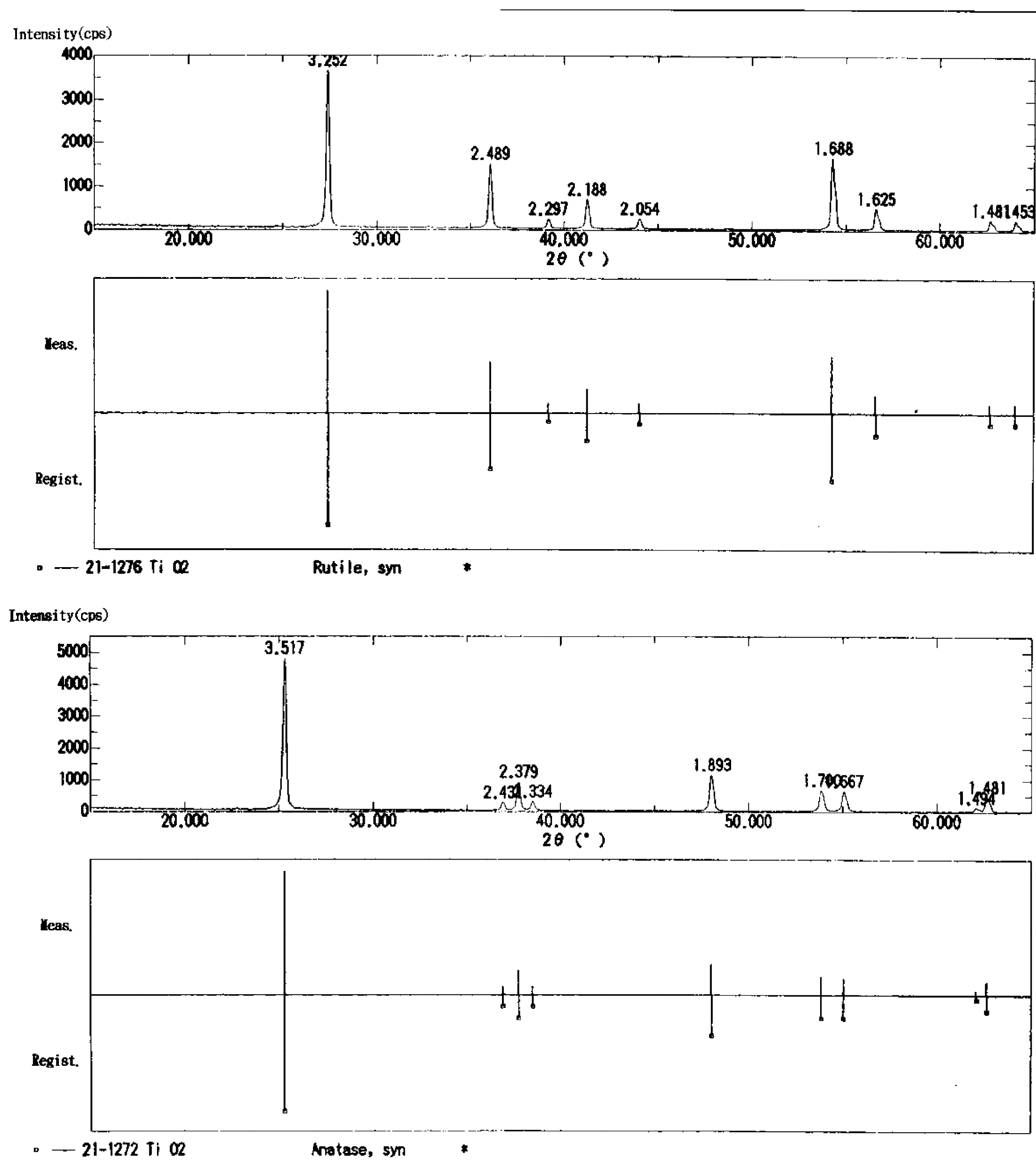


Fig. 4 Difference in diffraction pattern of TiO_2 (crystal).

Both of the samples in Fig. 3 are SiO_2. However, while a broad halo was observed with the upper-side quartz glass (amorphous), a sharp diffraction pattern was observed with the lowerside quartz (crystalline). In this way X-ray diffraction allows one to know if the sample is crystallized; and, if so, to what extent (crystallinity). Early use of XRD by the pharmaceutical industry involved the selection of medicine based on the crystalline or amorphous state of the material.

The samples in Fig. 4 are TiO_2, as they are expressed by chemical formula. One can determine from the sharp diffraction patterns that they are both crystalline. However, the difference in their diffraction pattern shows that their crystal structures are not the same. The use of qualitative XRD analysis clearly distinguishes the upper sample as rutile and the lower one as anatase. As is illustrated in these cases, X-ray diffraction permits the distinction of crystals through their distinct diffraction patterns.

Analyses that can be performed by the MiniFlex^{+}, and XRD in general, are summarized as follows:

1) Qualitative analysis (e.g., identification of rock minerals, crystals in various raw materials, and impurities can be determined from diffraction patterns)

2) Quantitative analysis (e.g., quantitation of asbestos, free silicic acid in particulate dust, and impurities is performed by integrating the intensities of diffraction lines)

3) Crystallinity, important in the analysis of polymers (the ratio of integrated intensities of crystal diffraction lines against the total scattered X-ray intensities is utilized)

4) Crystallite size (evaluation of the crystallite size of materials is determined from the diffraction line width). Cf. Degree of separation of the five fingers of quartz is also an evaluation of the quartz diffraction line width, from which the reactivity of alkali silica can be determined.

5) Lattice constant (evaluation of the element ratio, solid solubility, etc. is determined from a peak shift)

4. Specification & Features of the MiniFlex$^+$

Rigaku's general purpose powder diffraction XRD systems, the MiniFlex$^+$ and the D/max2000 series are computer controlled. The specification of the MiniFlex$^+$ and those of the D/max 2000 series are compared in Table 1 to highlight the MiniFlex$^+$ features.

The features of the MiniFlex$^+$ include the following.

Table 1 comparison of major specifications between MiniFlex$^+$ and general-purpose system (D/max-2000)

Item	MiniFlex$^+$	D/max-2000
X-ray output	450 W (with 1 kW tube as standard)	2000 W (with 2kW tube as standard)
Detector	Small scintillation counter	Scintillation counter
Goniometer type	Vertical	Vertical, horizontal, theta-theta type for selection
Goniometer radius	150 mm	185 mm
Optical system (including options)	Focusing method	Focusing method, thin film optics, etc
Slit	DS: variable slit RS: 0.3 mm fixed	Auto exchange fixed slit, variable slit system
Sample stage (including options)	Standard sample stage, specimen rotation attachment, sample changer	Standard sample stage, specimen rotation attachment, sample changer, high & low temp. attachments, etc.
Computer	Notebook type personal computer (PC), etc.	Work station (EWS)
OS/Window	Windows	UNIX/X-Window
Software (including options)	Peak search, qualitative, quantitative (calibration curve method, MF method, environmental particle dust quantitation, etc.). Integrated intensity calculation (peak position, width, etc., also), rock mineral software, etc.	Peak search, qualitative, quantitative (calibration curve method, MF method, environmental particle dust quantitation, etc.). Lattice constant refinement, crystallite size, crystallinity, pole figure, stress, RIETAN, etc.
Dimensions	560 W x 244 D x 582 H mm	1200 W x 1050 D x 1740 H mm
Weight	66 kg	600 kg
Power supply	220 V or 115 V/15 A	200 V/20 A, 100 V/35 A
Cooling water	Small water circulating pump for dedicated use (option). etc.	Various water circulating pumps (option). etc.

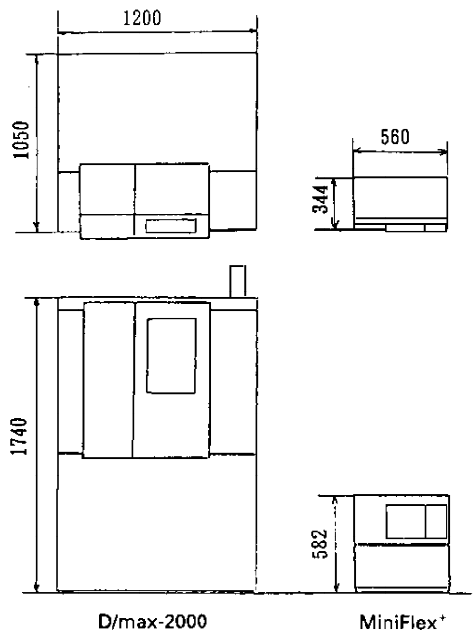


Fig. 5 Difference in size between MiniFlex$^+$ and D/max-2000.

1. Ultra Small Size; Extremely Light Weight,Easy Installation

Figure 5 shows the difference in size between the MiniFlex$^+$ and the D/max 2000; to install the D/max 2000, it is necessary to prepare a lab with the appropriate space. Installation of the MiniFlex$^+$ requires no lab space preparation and no more room than a desktop PC. The MiniFlex$^+$ design, with its small size and light weight, is perfect for transporting to the field (Fig. 6). As a full scale X-ray diffractometer equipped with a data processing function, the MiniFlex$^+$ is the smallest and the lightest in the world.

The MiniFlex$^+$ allows installation anywhere and earth grounded 220V or 110V power supply is available. It can be installed near a production line to provide quality control of materials or products; it can even be taken to a geological survey field office to identify minerals. The MiniFlex$^+$ can readily be placed near the sample collection area for on-site analysis. It has eliminated the need to send a sample to the lab and to wait to receive the final results. Such immediate analysis results obviously allow increased work efficiency.

2. Plug 'n Play XRD

The MiniFlex$^+$ uses a standard X-ray tube. A Cu tube, or any of the other available tube types, is factory installed and adjusted prior to shipment. A specimen rotation attachment and a sample changer are provided as options in addition to the standard sample stage. Replacement is simple and there is no need for alignment after replacement. Unlike the conventional XRD systems, the MiniFlex$^+$allows the operator to begin measurements without additional adjustments.

Fig. 6 MiniFlex$^+$ carried in Station Wagon.

This Plug 'n Play concept was developed for the MiniFlex$^+$ in order to eliminate the need for system adjustments. Its design philosophy differs from that of conventional XRD systems, as the MiniFlex$^+$ incorporated as automatic axial adjustment system. This is a key difference between this field oriented unit and conventional lab equipment.

3. Safety

The MiniFlex$^+$ is designed with its metallic housing interior used as the X-ray control section; the measurement chamber is completely isolated from the operator. Also, the sample chamber door is interlocked with the mechanical shutter of the X-ray window to ensure that when the door is open, the shutter will close. This allows maximum safety and prevents exposure in the remote possibility of erroneous operation.

4. Windows Operating System

A personal computer running on a Windows platform controls the MiniFlex+ measurement and data processing. Either a notebook type and/or a desktop type PC can be used. The screen operation is easy and the software enables the operator to conduct measurements and to process data without the need of in-depth diffraction knowledge.

5. Cost/Performance

The MiniFlex+ incorporates many sophisticated components, such as a vertical goniometer where one stepping motor scans two axes. An automatic divergence function (patent pending) is standard, as is the newest high frequency type X-ray generator and a newly developed small sized scintillation counter. These innovative technologies have resulted in a substantially more compact size and a price approximately 1/3 less than that of conventional powder diffractometers.

The cost merits are not limited to the price of the product. Also reduced are the electrical costs, the required installation area, the overall operation costs, etc. Overall, the MiniFlex+ may be said to be a diffractometer with an excellent cost/performance ratio. The system offers a relatively easy purchase even for small scale labs.

As an example, the MiniFlex+ is ideally suited for materials characterization experiments by students because multiple units can be installed for around the same cost as one general-purpose conventional XRD system. The MiniFlex+ is also a good complement to labs with X-ray Fluorescence analysis capabilities.

6. Fundamental Performance of the MiniFlex+

Although the MiniFlex+ X-ray generator is 450 watts of power, actual samples we have measured for customers exhibit good detection for trace components and satisfactory peak width evaluations.

1. Typical Scanning Parameters

For most samples analyzed, a scanning speed of 2°per minute was used; qualitative measurements took about 30 minutes per sample (angular region of $3° \leq 2\theta \leq 70°$). As mentioned, test measurements were conducted on a number of samples and analyzed for qualitative information. The date collected had sufficient intensities to allow excellent phase identification. This was also the case when measuring paper coating materials. From the study, it was deduced that a full range scan in a 30 minute time period will result in intensities sufficient for qualitative analysis. This would be true unless the samples were of poor crystallinity which could be caused by humidity or some other phenomenon.

For detection of small peaks or evaluation of peak shapes. the scan speed was chosen based on intensity. This type of measurement requires a much shorter angular range, perhaps as small as 2 to 3°. Therefore, even if it is necessary to slow the scan speed to achieve acceptable counting statistics, the overall required time should not be significant. The MiniFlex+ been able to sufficiently detect 1% asbestos (tremolite) and 0.3% free silicic acid (αSiO_2) contained in particulate dust.

The variable divergence slit keeps the area of irradiation constant on the sample regardless of the $\theta/2\theta$ angle. This will give better intensity at the higher 2θ range when compared to a fixed slit system. As expected, with higher intensity comes a slight decrease in the resolution, but when using the data for qualitative analysis this slight decrease is transparent. Surprisingly, there have been cases in which the intensity of data obtained with the MiniFlex+ has been higher than data collected on the same sample with a conventional XRD system.

2. Resolution

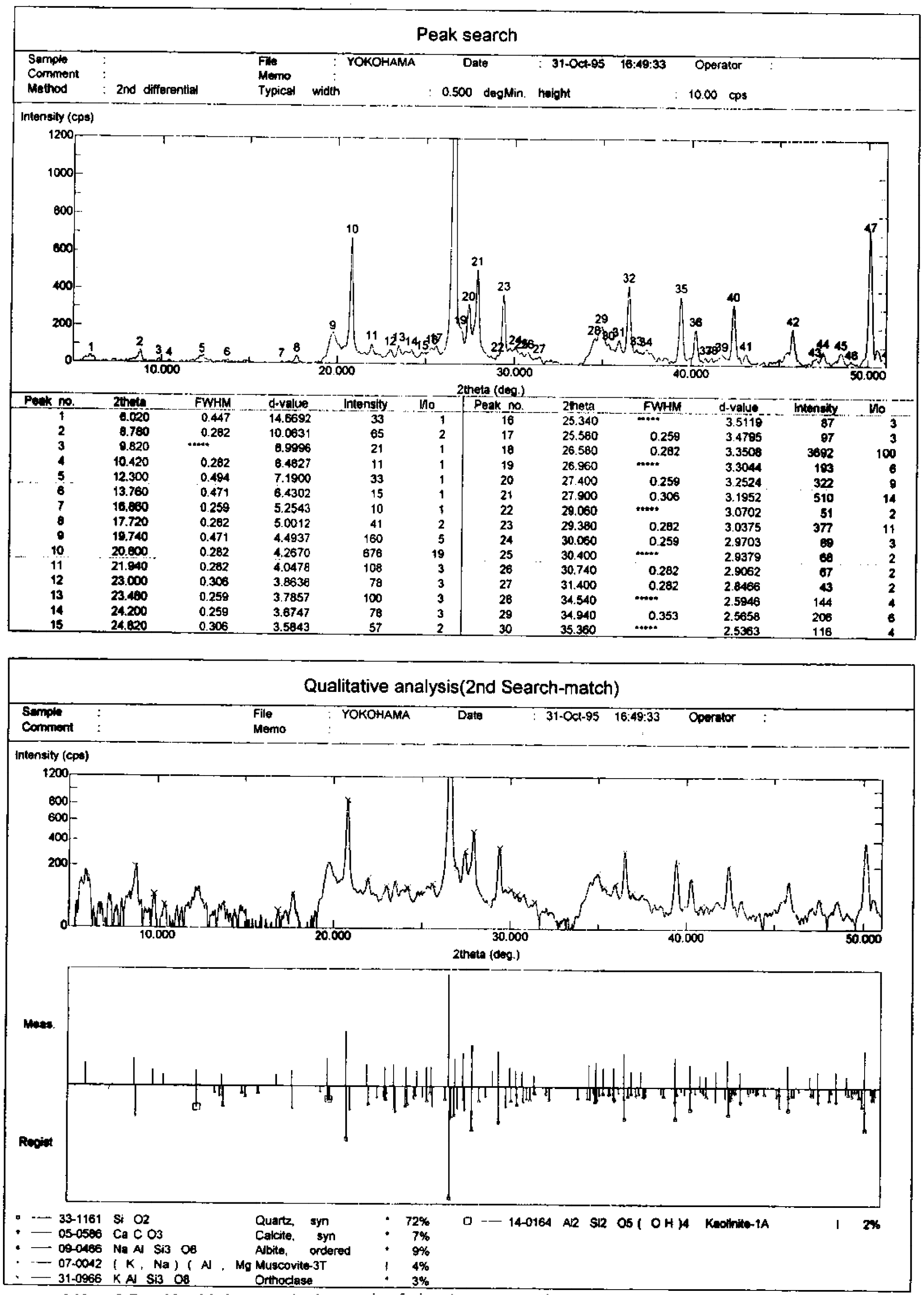
Peak resolution was not sacrificed in designing this compact MiniFlex+. With the smaller radius of the goniometer (150mm radius), one might expect less resolution overall; and, since the receiving slit is fixed, one cannot improve resolution by using a finer receiving slit. However, when the five fingers of quartz measured on the MiniFlex+ is compared with that measured on a standard X-ray diffractometer, there is a good correlationship between both data. In other words, the MiniFlex+ has sufficient resolution as it is.

Using a fine focus tube, which has a focal width of 0.4 mm rather than the 1 mm width of a normal focus tube, will further improve the resolution. Even though the fine focus tube has less output than the normal focus tube, it is recommended to use the fine focus type if an increase in resolution is the desired effect.

7. MinFlex+ Applications Data

The demonstrations we have given of the MiniFlex+ have invariable impressed the viewers. Its actual size is smaller than that perceived from the pictures and the brochures. All have been extremely surprised at the high quality of data which can be collected in such a short time.

Figure 7 shows the qualitative analysis results of few sample types.



Peak no.	2theta	FWHM	d-value	Intensity	I/Io	Peak no.	2theta	FWHM	d-value	Intensity	I/Io
1	6.020	0.447	14.6692	33	1	16	25.340	*****	3.5119	87	3
2	8.780	0.282	10.0631	65	2	17	25.580	0.259	3.4795	97	3
3	9.820	*****	8.9996	21	1	18	26.580	0.282	3.3508	3692	100
4	10.420	0.282	8.4827	11	1	19	26.960	*****	3.3044	193	6
5	12.300	0.494	7.1900	33	1	20	27.400	0.259	3.2524	322	9
6	13.760	0.471	6.4302	15	1	21	27.900	0.306	3.1952	510	14
7	16.860	0.259	5.2543	10	1	22	29.060	*****	3.0702	51	2
8	17.720	0.282	5.0012	41	2	23	29.380	0.282	3.0375	377	11
9	19.740	0.471	4.4937	160	5	24	30.060	0.259	2.9703	89	3
10	20.800	0.282	4.2670	676	19	25	30.400	*****	2.9379	68	2
11	21.940	0.282	4.0478	108	3	26	30.740	0.282	2.9062	67	2
12	23.000	0.306	3.8636	78	3	27	31.400	0.282	2.8466	43	2
13	23.480	0.259	3.7857	100	3	28	34.540	*****	2.5946	144	4
14	24.200	0.259	3.6747	78	3	29	34.940	0.353	2.5658	206	6
15	24.820	0.306	3.5843	57	2	30	35.360	*****	2.5363	116	4

Fig 7.1 Qualitative analysis result of cla (upper: peak search, lower: search/match result).

8. Summary

Unlike conventional powder X-ray diffractometers, the MiniFlex^{+} gives the impression of being integral to the scientist. It has a novel shape, high performance. good response and provides quality data.

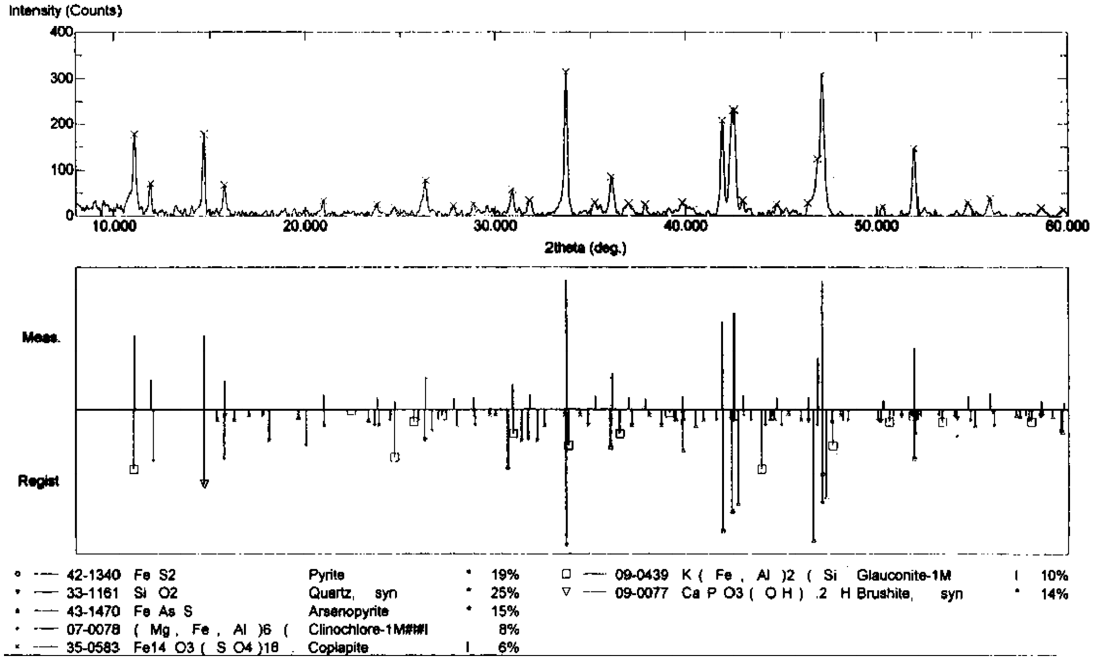


Fig. 7.2 Qualitative analysis of iron ore.

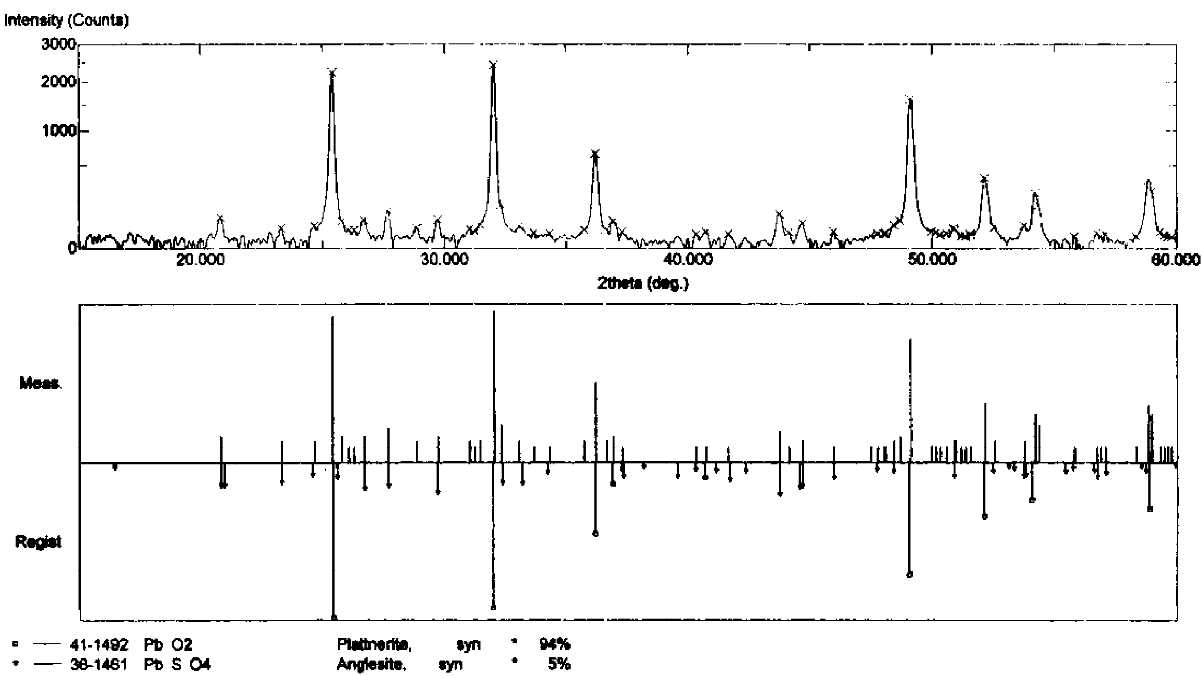


Fig 7.3 Qualitative analysis result of battery material. (It can be known that trace $PbSO_4$ is contained.)

The MiniFlex$^+$ was first announced in 1995, the year that commemorated the 100th year anniversary of the discovery of X-ray by Roentgen. Rigaku continues to be the leader in the development of X-ray equipment and in the expansion of practical applications of X-ray technology. Rigaku puts X-rays to work the world over to keep our customers at the forefront of their technology.