

THERMOGRAVIMETRIC AND INFRARED SPECTRAL STUDIES FOR RAPID EVALUATION OF ROCK PHOSPHATES

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Thermal, infrared spectral and chemical analyses have been carried out on fourteen rock phosphate samples from five deposits in Hazara, North West Frontier Province (NWFP), Pakistan. Samples with high phosphate content have shown less than 3% loss in weight on ignition and a set of two characteristic medium intensity absorption bands in the range 1460-1420 cm^{-1} . On the other hand ores samples with low phosphate content have shown high loss in weight on ignition and only one absorption band around 1460-1420 cm^{-1} in their infrared spectra. The analytical data described and discussed in this paper suggests that TGA/DTA and infrared spectral techniques may be used as a rapid means for the preliminary selection of ore-grades, in place of conventional techniques.

Key words: Phosphates, Infrared, Thermogravimetry.

Introduction

A number of standard procedures and analytical methods are known and used extensively for sample preparation and determination of P_2O_5 content in phosphate ores [1-3]. In the acid decomposition procedure the ore is treated with hydrochloric acid or a mixture of HCl and nitric acid or perchloric acid. In the fusion decomposition method the sample under study is fused in a platinum crucible using anhydrous sodium carbonate as a flux, followed by digestion with concentrated hydrochloric acid. P_2O_5 content in the sample solution, so obtained by either of the decomposition methods, may be determined (i) gravimetrically by precipitating the phosphate as phosphomolybdate or magnesium pyrophosphate or (ii) by dissolving the phosphomolybdate precipitate in a known volume of standard sodium hydroxide solution and back-titrating it with standard nitric acid solution or (iii) colorimetrically using a suitable colour developing reagent such as 1-amine-2-naphthol-4-sulphonic acid, molybdate hydrazine sulphate or ammonium vanadate [1-3]. However, all these methods are laborious, tedious, need expensive chemicals and are time consuming. In the present investigations TGA/DTA and IR studies, carried out on the fourteen samples of rock phosphates from five deposits in Hazara (NWFP), have shown that TGA/DTA and IR techniques may be used for evaluation of the phosphate ore-grades in a short period of time. Details of these investigations and results achieved are described and discussed in this paper.

Petrological and geochemical studies of some Hazara (NWFP) phosphates deposits have been reported in details by a number of workers. The early Cambrian phosphate deposits of Hazara occur as discontinuous outcrops and in the form of

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lenses. These are hosted in Galdanian member of the Abbotabad formation and occur in association with phosphatic dolomite and dolomite with distorted horizons of chert. Generally, these are of two types; microphosphorites (finely crystalline anthigenic mud) and oval shaped pellets. Mineralogically, Hazara phosphates have been identified generally as hydroxylapatite and fluorapatite [4-5].

Experimental

Samples of phosphate rocks were collected from Lambidogi, Bataklnala, Lagarban, Kakul and Tarnawai. Representative powdered samples (-100 mesh) were used throughout for TGA/DTA, infrared and chemical analysis.

Thermal analyses. Thermogravimetric and differential thermal analyses (TGA & DTA) of samples weighing 20-25 mg were carried out in platinum boat, at a heating rate of 10°/min under inert atmosphere (purified nitrogen) and a gas flow rate of 50 ml/min on a Dupont TGA-model 951 with temperature range 25-1000°, coupled with a Dupont 1090 computer controlled thermal analyzer and controller.

Infrared spectra. Spectra were recorded on a Hitachi model-270-50 infrared spectrophotometer in the range 4000-250 cm^{-1} using KBr disc techniques.

Chemical analyses. Sample solution preparation was carried out by both the fusion and acid decomposition methods. In the fusion decomposition, the powdered phosphate rock samples were fused in a platinum crucible using anhydrous sodium carbonate as a flux followed by digestion with concentrated hydrochloric acid. In the acid decomposition, the powdered phosphate rock was treated with HCl (conc.) followed by HCl (conc.) and HNO_3 (1:4) mixture and treatment with HClO_4 (70-77%). P_2O_5 content in the sample

solution was determined by (i) gravimetric method, first separating as phosphomolybdate followed by precipitation as magnesium ammonium phosphate hexahydrate, using magnesia mixture and (ii) spectrophotometric method using vanadomolybdate reagent and measuring the intensity of the resulting yellow-coloured complex at 450 nm on EPMA model LS-7 spectrophotometer [1-3].

Results and Discussion

The analytical data from DTA/TGA, IR studies and chemical analyses are described in Tables 1 to 4. Thermograms (TG) and partial IR spectra of a few samples are also shown in Figs. 1-3. TG and IR spectra of samples I, II and IX, X are not reproduced here as these have already been given in our earlier reports on geochemical studies of Lambidogi phosphates [4-5].

Thermogravimetric and differential thermal analyses (TGA & DTA) studies. From comparison of TG of phosphate rock samples studied, total weight loss for some samples was

TABLE 1. DIFFERENTIAL THERMAL AND THERMOGRAVIMETRIC (DTA & TGA) ANALYSES OF HIGH-GRADE HAZARA (NWFP) ROCK PHOSPHATES.

Sample No.	Deposit location	Decomposition temperatures(°C)	Total weight loss (%)
I	Lambidogi	730 - 980	1.50
II	Lambidogi	730 - 980	2.77
III	Batakna	556 - 764	2.85
IV	Batakna	575 - 710	2.44
V	Kakul	120 - 650*	0.86
VI	Kakul	120 - 650*	1.23
VII	Tarnawi(Eastern)	557 - 712	2.73
VIII	Tarnawi(Eastern)	561 - 704	2.93

* Slow constant loss.

TABLE 2. DIFFERENTIAL THERMAL AND THERMOGRAVIMETRIC ANALYSES (DTA & TGA) OF LOW-GRADE HAZARA (NWFP) ROCK PHOSPHATES.

Sample No.	Deposit location	Decomposition temperatures(°C)	Total weight loss (%)
IX	Lambidogi	660-820(730) 830-980(920)	41.25
X	Lambidogi	660-840(770) 850-980(920)	20.00
XI	Lagarban	698-780(760)	33.79
XII	Lagarban	707-780(760)	42.60
XIII	Lagarban	698-780(760)	33.95
XIV	Kakul	667-765(730)	16.34

Figures in parenthesis are peak temperatures.

found to be very small as compared to others (Tables 1, 2). Samples I to VIII indicated weight loss of less than 3%. Samples V and VIII showed the minimum and maximum weight loss of 0.86% and 2.93%, respectively (Table I). On the other hand, samples IX to XIV showed significantly greater weight loss, the maximum being 41.25% and the minimum 16.34% for samples IX and XIV, respectively (Table 2). TG of samples I to VIII showed similar thermal characteristics, generally a slow constant weight loss over a large temperature range between 550-750°. This weight loss may be attributed to loss of water, phosphorous oxyfluoride or chlorine from hydroxy-, fluoro- or chloro-apatites, respectively [6]. The weight loss in this temperature range may also be possibly caused by the oxidation of organic matter or decomposition of the carbonate moiety of the small amount of host rock associated with the apatite [7]. It has been revealed by spectral studies that carbonate may be present in the form of CO₃²⁻ ions replacing PO₃³⁻ in the apatite crystals [7]. For samples V and VI the weight loss appeared to start at a much lower temperature of 120° which may be due to escape of differently bound water [7]. An increase in mass around 850°

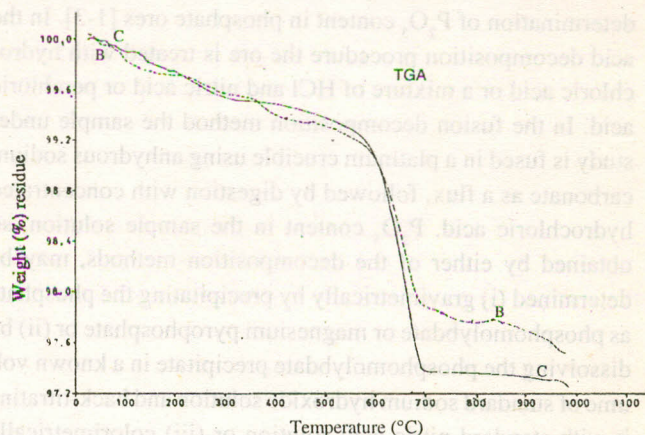


Figure I(a). Thermogravimetric analyses (TGA) curves B and C for samples IV and VII respectively.

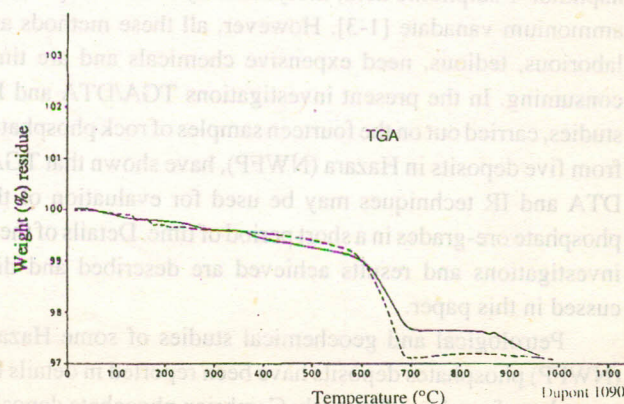


Figure I(b). Thermogravimetric analyses (TGA) curves A and D for samples III and VIII respectively.

(Fig. 1b) may be caused by oxidation of minor metals in the rock samples.

No characteristic DTA peak was observed in TG of samples I to VIII. Among samples IX to XIV, only Lambidogi samples IX and X showed thermal characteristics different from the other samples (Table 2). Samples XI to XIV showed only one endothermic peak corresponding to one stage of thermal decomposition in the temperature range 660-780°

(endothermic peak temperature for samples XII-XIII = 760°; sample XIV = 730°). However, samples IX and X also showed an additional endothermic peak around 920° indicating a second stage of thermal decomposition in the temperature range 830-980° (Table 2). A number of workers (5, 8-10) have attributed the weight loss in these temperature ranges to the CO₃ decomposition of calcite (CaCO₃) or of CO₃ of dolomite [MgCa(CO₃)₂] structures according to the following

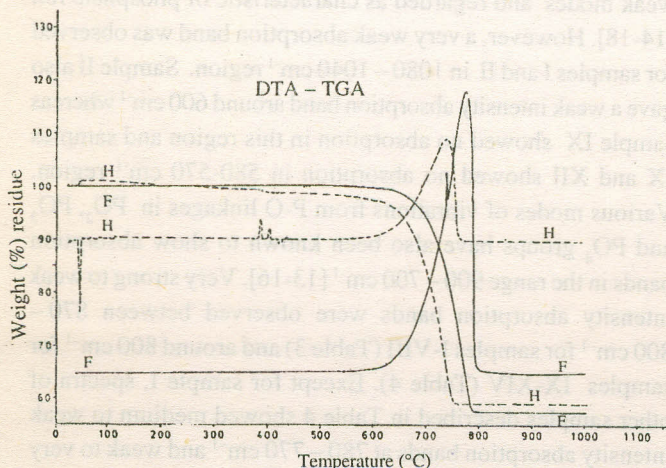


Fig. 2(a). Thermogravimetric and differential thermal analyses (TGA & DTA) curves F and H for samples XII and XIV respectively.

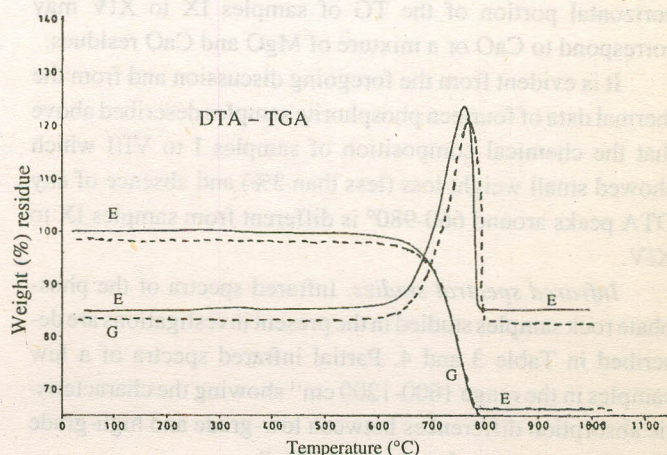


Fig. 2(b). Thermogravimetric and differential thermal analyses (TGA & DTA) curves E and G for samples XI and XIII respectively.

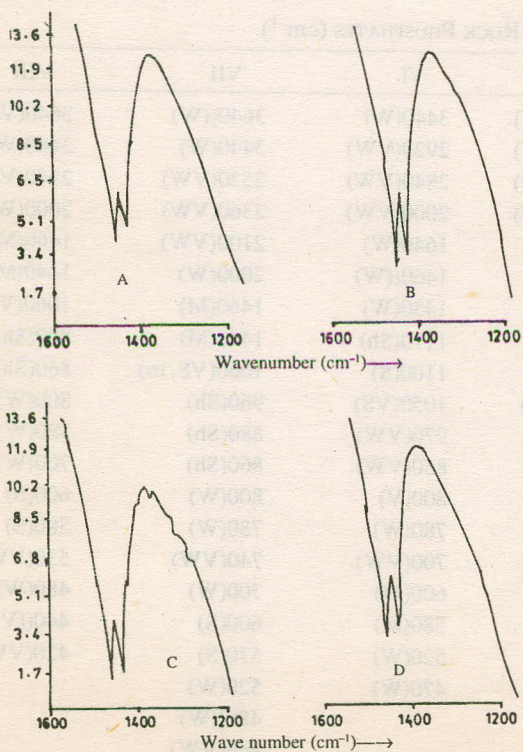


Fig. 3(a). Partial infrared spectra of high-grade phosphate ores. Curves A,B,C and D for samples III, IV, VII and VIII respectively.

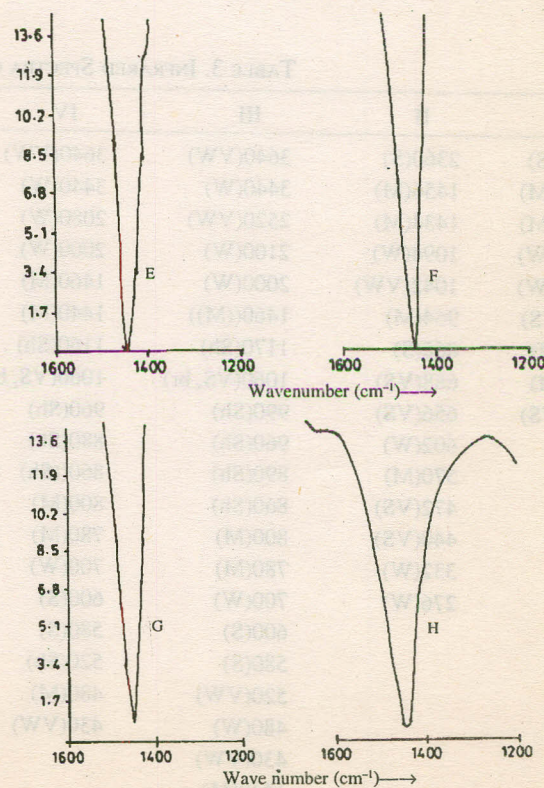
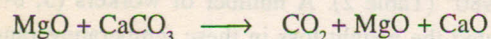
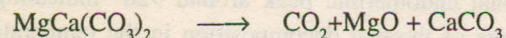


Fig. 3(b). Partial infrared spectra of low-grade phosphate ores. Curves E,F,G and H for samples XI,XII,XIII and XIV respectively.

chemical reactions:



The decomposition was completed around 980° as no further loss in weight was observed beyond this temperature. The horizontal portion of the TG of samples IX to XIV may correspond to CaO or a mixture of MgO and CaO residues.

It is evident from the foregoing discussion and from the thermal data of fourteen phosphorite samples described above that the chemical composition of samples I to VIII which showed small weight loss (less than 3%) and absence of any DTA peaks around 660-980° is different from samples IX to XIV.

Infrared spectral studies. Infrared spectra of the phosphate rock samples studied in the present investigations are described in Table 3 and 4. Partial infrared spectra of a few samples in the range 1600-1200 cm⁻¹ showing the characteristic absorption differences between low-grade and high-grade phosphate ores are also shown in Fig.3.

A number of workers have described and discussed the infrared spectra of phosphorous compounds and have

suggested the assignments of characteristic absorption bands to the various structural features of such compounds [11-13]. Recently, Banerjee *et al.* have reported infrared spectra of some phosphate ores of Udaipur and Jhabua in India [14]. In the present studies infrared absorption spectra of Hazara phosphate ores under investigations showed absorption bands in the regions 1080 – 1040 cm⁻¹, 600 cm⁻¹ and 580 – 570 cm⁻¹ thought to be resulting from P-O bonding and P-O weak modes and regarded as characteristic of phosphate ion [14-18]. However, a very weak absorption band was observed for samples I and II in 1080 – 1040 cm⁻¹ region. Sample II also gave a weak intensity absorption band around 600 cm⁻¹ whereas sample IX showed no absorption in this region and samples IX and XII showed no absorption in 580-570 cm⁻¹ region. Various modes of vibrations from P-O linkages in PO₂, PO₃ and PO₄ groups have also been known to show absorption bands in the range 900 – 700 cm⁻¹ [13-16]. Very strong to weak intensity absorption bands were observed between 870 – 800 cm⁻¹ for samples I-VIII (Table 3) and around 800 cm⁻¹ for samples IX-XIV (Table 4). Except for sample I, spectra of other samples described in Table 4 showed medium to weak intensity absorption bands at 780 – 770 cm⁻¹ and weak to very weak absorption bands at 700 cm⁻¹ assigned to O-P-O linkages [13]. Sample II showed a very strong absorption band at 688

TABLE 3. INFRARED SPECTRA OF HIGH-GRADE ROCK PHOSPHATES (cm⁻¹).

I	II	III	IV	V	VI	VII	VIII
2926(S)	2360(S)	3640(VW)	3640(VW)	3420(VW)	3440(W)	3640(W)	3640(VW)
1458(M)	1456(M)	3440(W)	3440(W)	2920(VW)	2920(VW)	3440(W)	3440(W)
1434(M)	1434(M)	2520(VW)	2080(W)	2840(VW)	2840(VW)	2520(VW)	2540(VW)
1095(W)	1094(W)	2100(W)	2000(W)	2000(VW)	2000(VW)	2360(VW)	2000(W)
1044(W)	1042(VW)	2000(W)	1460(M)	1640(W)	1640(W)	2100(VW)	1460(M)
864(VS)	964(M)	1460(M)	1440(M)	1450(W)	1460(W)	2000(W)	1440(M)
603(M)	866(S)	1170(Sh)	1160(Sh)	1420(W)	1430(W)	1460(M)	1060(VS, br)
573(M)	688(VS)	1060(VS, br)	1060(VS, br)	1160(Sh)	1170(Sh)	1440(M)	880(Sh)
474(VS)	656(VS)	990(Sh)	960(Sh)	1100(M)	1100(S)	1060(VS, br)	860(Sh)
	602(W)	960(Sh)	880(Sh)	1040(VS)	1050(VS)	960(Sh)	800(W)
	570(M)	890(Sh)	860(Sh)	960(VW)	970(VW)	880(Sh)	780(W)
	472(VS)	860(Sh)	800(M)	870(VW)	860(VW)	860(Sh)	700(W)
	440(VS)	800(M)	780(M)	800(W)	800(V)	800(W)	600(S)
	332(W)	780(M)	700(W)	770(W)	780(W)	780(W)	580(S)
	276(W)	700(W)	600(S)	700(VW)	700(VW)	740(VW)	530(VW)
		600(S)	580(S)	600(M)	600(M)	700(W)	480(W)
		580(S)	520(Sh)	570(M)	580(M)	600(S)	440(VW)
		520(VW)	480(M)	470(W)	520(W)	570(S)	420(VW)
		480(W)	430(VW)		470(W)	520(W)	
		430(VW)				480(W)	
		1440(M)				440(VW)	
						420(VW)	

VS = Very strong. S = Strong. M = Medium. W = Weak. VW = Very weak. Sh = Shoulder. br = broad.

cm⁻¹. However, some workers [14] have assigned absorption bands in the range 870-710 cm⁻¹ to vibrations resulting from C-O linkages of carbonate of the associated dolomite or calcite present in apatites (carbonate fluor-apatites). The absorption bands shown in the range 4650-2500 cm⁻¹ in the spectra of samples I-XIV have been assigned to vibrations resulting from O-H, P-H, P-O-H or H-bonded O-H linkages [11,15-17].

A comparison of the infrared spectra of samples I-VIII (Table 3) with those of samples IX to XIV (Table 4) showed distinct spectral differences in the region 1460-1420 cm⁻¹ which, as will be discussed later, appeared to be of some analytical importance (Fig.3). Samples IX to XIV showed only one absorption band of varying intensity around 1450 cm⁻¹. On the other hand, samples I to VIII showed a set of two absorption bands mostly of medium intensity around 1460 cm⁻¹ and around 1440 cm⁻¹. Samples V and VI showed the second absorption band at 1420 cm⁻¹ and 1430 cm⁻¹ respectively. From the infrared spectral study of twenty phosphorites ores samples, a strong absorption doublet in the range 1450-1440 cm⁻¹ has been reported by Banerjee *et al.* [14] who considered it to be characteristics of phosphorites concentrates. Both the infrared spectral and X-ray diffraction studies of phosphate ores have indicated the presence of dolomite/calcite in discrete phases mixed with apatite. In some

phosphate ores the partial substitution of PO₄ by CO₃ have also been reported to be more pronounced than others [14].

From the foregoing discussion it may be inferred that the characteristic spectral difference observed in the infrared region 1460-1420 cm⁻¹ may be due to the relative concentrations of phosphate content in the ores which is supported by the chemical analyses of P₂O₅ content of the samples I-XIV (Table 5). The analytical data described in Table 5 clearly indicate that samples showing less than 3% loss in weight on ignition and a set of two medium intensity absorption bands in the range 1460-1420 cm⁻¹ in their infrared absorption spectra, appear to be high-grades rock phosphates (samples I-VIII) with P₂O₅ content greater than 30%. On the other hand those samples which show large loss in weight on ignition and do not show a set of two absorption bands in the range 1460-1420 cm⁻¹ in their infrared spectra may be regarded as poor-grade rock phosphates (samples IX to XIV) with low P₂O₅ content (<30%). Out of fourteen samples studied in the present investigations, only sample XIV showed some discrepancy. However, it is suggested that weight losses between 10-20% must be considered with caution and for such samples, the intensity of other P-O absorption bands at 1000-1200 cm⁻¹ and/or 570-600 cm⁻¹ in their infrared absorption spectra, should also be taken into consideration.

TABLE 4. INFRARED SPECTRA OF LOW-GRADE ROCK PHOSPHATES (cm⁻¹).

LD-IX	LD-X	LG-XI	LG-XII	LG-XIII	KK-XIV
2924(S)	2928(M)	3440(W)	3440(W)	3440(W)	3440(W)
2540(S)	2532(S)	3000(VW)	3000(VW)	3000(VW)	2920(VW)
1828(M)	1448(W)	2880(VW)	2880(VW)	2840(VW)	2520(VW)
1440(VW)	1092(W)	2520(VW)	2620(VW)	2620(VW)	2000(VW)
1096(M)	1060(W)	1840(VW)	2520(VW)	2520(VW)	1820(VW)
1048(M)	880(M)	1450(VS)	1820(VW)	1820(W)	1640(W)
880(W)	798(S)	1170(VW)	1450(VS)	1450(VS)	1450(S)
728(M)	780(S)	1080(W)	1080(W)	1170(VW)	1100(M)
472(S)	728(S)	880(M)	1060(W)	1100(M)	1040(VS)
370(W)	694(VS)	790(W)	880(M)	1040(M)	960(VW)
322(M)	600(S)	730(W)	780(W)	880(M)	880(W)
264(M)	572(S)	690(VW)	730(W)	870(Sh)	790(W)
	516(S)	600(VW)	600(VW)	800(W)	730(W)
	470(M)	580(VW)		780(W)	600(M)
	372(W)	510(VW)		730(W)	580(M)
	320(M)	460(W)		600(W)	470(VW)
	262(M)			570(W)	
				510(W)	
				460(W)	

VS= Very strong. S=Strong. M = Medium. W= Weak. VW=Very weak. Sh = Shoulder.

Conclusion

High-grade phosphate ores have indicated minor losses in weight (<3%) on ignition as compared to low-grade

TABLE 5. COMPARATIVE LOSS ON IGNITION, IR ABSORPTION BANDS AND P₂O₅ CONCENTRATIONS OF HAZARA (NWFP) ROCK PHOSPHATES.

Sample No.	Total loss in weight (%)	IR absorption bands (cm ⁻¹)	P ₂ O ₅ content (%)
I	1.50	1458(M);1434(M)	36.20
II	2.77	1456(M);1434(M)	39.03
III	2.85	1460(M);1440(M)	31.49
IV	2.44	1460(M);1440(M)	31.73
V	0.86	1450(W);1420(W)	38.07
VI	1.23	1460(W);1430(W)	35.00
VII	2.73	1460(M);1440(M)	30.18
VIII	2.93	1460(M);1440(M)	30.18
IX	41.25	1440(VW)	16.69
X	20.00	1448(W)	11.02
XI	33.79	1450(VS)	Not detected
XII	42.60	1450(VS)	Traces
XIII	33.95	1450(VS)	7.98
XIV	16.34	1450(S)	28.25

M = Medium. W = Weak. VW = Very weak. VS = Very strong. S = strong.

phosphate ores. In the infrared spectra of high-grade phosphate ores, a set of two medium intensity absorption bands is observed in the range 1460–1420 cm⁻¹ compared to one absorption band in this range shown by low-grade phosphate ores.

On the basis of above observations, thermogravimetric analyses and infrared spectral data may be used for a preliminary selection and evaluation of phosphate ores grades which may be followed by routine chemical analyses of high-grade ores, thus selected, for confirmation of P₂O₅ content.

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Conclusion

High grade phosphate ores have indicated mineral losses in weight (%) on routine as compared to low-grade

TABLE 2. Comparative IR and TGA on Routine IR Absorption Bands and P₂O₅ Concentrations of Various (W/W) Rock Phosphates

Sample No.	Total loss in weight (%)	IR absorption bands (cm ⁻¹)	P ₂ O ₅ content (%)
I	1.20	1420(W), 1430(M)	30.20
II	1.77	1420(W), 1430(M)	29.03
III	2.57	1420(W), 1430(M)	31.49
IV	2.44	1420(W), 1430(M)	31.33
V	0.98	1420(W), 1430(W)	38.2
VI	1.33	1420(W), 1430(W)	32.00
VII	2.51	1420(W), 1430(M)	30.18
VIII	2.93	1420(W), 1430(M)	30.18
IX	4.25	1420(W)	34.60
X	30.00	1420(W)	31.02
XI	27.75	1420(W)	30.00
XII	43.00	1420(W)	30.00
XIII	37.82	1420(W)	30.00
XIV	18.24	1420(W)	38.22

TABLE 4. Infrared Spectra of Low-Grade Rock Phosphates (part 2)

IR (cm ⁻¹)	IR (cm ⁻¹)	IR (cm ⁻¹)	IR (cm ⁻¹)	IR (cm ⁻¹)	IR (cm ⁻¹)	IR (cm ⁻¹)	IR (cm ⁻¹)	IR (cm ⁻¹)	IR (cm ⁻¹)
1420(W)	1430(M)	1420(W)	1430(M)	1420(W)	1430(M)	1420(W)	1430(M)	1420(W)	1430(M)
1420(W)	1430(M)	1420(W)	1430(M)	1420(W)	1430(M)	1420(W)	1430(M)	1420(W)	1430(M)
1420(W)	1430(M)	1420(W)	1430(M)	1420(W)	1430(M)	1420(W)	1430(M)	1420(W)	1430(M)
1420(W)	1430(M)	1420(W)	1430(M)	1420(W)	1430(M)	1420(W)	1430(M)	1420(W)	1430(M)
1420(W)	1430(M)	1420(W)	1430(M)	1420(W)	1430(M)	1420(W)	1430(M)	1420(W)	1430(M)
1420(W)	1430(M)	1420(W)	1430(M)	1420(W)	1430(M)	1420(W)	1430(M)	1420(W)	1430(M)
1420(W)	1430(M)	1420(W)	1430(M)	1420(W)	1430(M)	1420(W)	1430(M)	1420(W)	1430(M)
1420(W)	1430(M)	1420(W)	1430(M)	1420(W)	1430(M)	1420(W)	1430(M)	1420(W)	1430(M)
1420(W)	1430(M)	1420(W)	1430(M)	1420(W)	1430(M)	1420(W)	1430(M)	1420(W)	1430(M)
1420(W)	1430(M)	1420(W)	1430(M)	1420(W)	1430(M)	1420(W)	1430(M)	1420(W)	1430(M)

W = Weak, M = Medium, V = Very strong, S = strong