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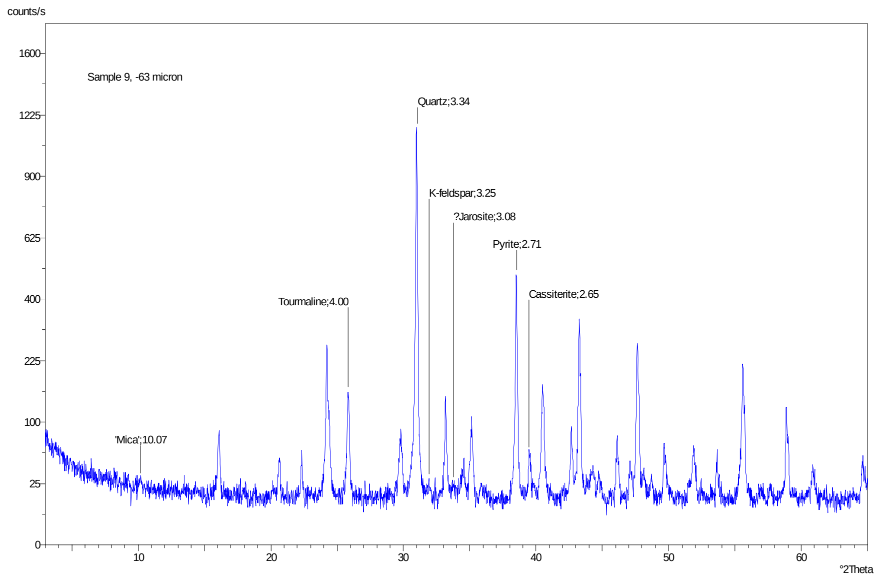
X-ray diffraction analysis of soil samples from abandoned mine sites in Cornwall (Part 2)

Environmental Protection Programme

Internal Report IR/03/013



BRITISH GEOLOGICAL SURVEY

INTERNAL REPORT IR/03/013

X-ray diffraction analysis of soil samples from abandoned mine sites in Cornwall (Part 2)

J A McKervey

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X-ray diffraction, Cornwall, mines, soil samples.

Front cover

XRD trace for the soil sample 9, -63µm.

Bibliographical reference

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1 Introduction

This report presents the results of qualitative X-ray diffraction analysis (XRD) analysis conducted for the project 'Fate and Transport of Heavy Metals'. The object of this project is to examine the environmental transport and fate of heavy metals, particularly in relation to abandoned mining and mineral processing operations. A set of soil samples separated into various size fractions, and into light and heavy fractions, was received from the BGS sample preparation facility and the details are listed in Table 1. These samples have previously be the subject of a whole-rock XRD study (McKervey, 2002).

Table 1: Sample list

Sample	Fraction	MPL code
9	-63 µm	MPLJ295
9	light minerals	MPLJ296
14	+32-63 µm	MPLJ297
16	heavy minerals	MPLJ298
18	heavy minerals	MPLJ299
19	+125 µm heavy minerals	MPLJ300
19	+63-125 µm heavy minerals	MPLJ301
19	+32-63 µm heavy minerals	MPLJ302
20	+125 µm heavy minerals	MPLJ303
20	+63-125 µm heavy minerals	MPLJ304
20	+32-63 µm heavy minerals	MPLJ305

2 Sample preparation

The samples were hand-ground in a pestle and mortar and then loaded using a drop of acetone onto a zero-background silicon wafer. For one sample (Sample 9, -63µm) sufficient ground material was provided to allow a full powder preparation. Approximately 3 g of the powder was micronised under acetone for 10 minutes to provide a finer and more uniform particle size and the powder was then back-loaded into a standard aluminium sample holder ready for XRD analysis.

3 XRD analysis

XRD analysis was carried out using a Philips PW1700 series diffractometer fitted with a cobalt-target tube and operated at 45 kV and 40 mA. The samples were scanned from 3-65 °2θ at 0.7 °2θ/minute. Diffraction data were analysed using Philips X'Pert software coupled to an International Centre for Diffraction Data (ICDD) database running on a Gateway personal computer system.

4 Results

The results of qualitative XRD analysis are given in Table 2 where a ranking (major (mj), minor (mi) or trace (tr)) is provided on the basis of relative X-ray intensity. A labelled XRD trace for each sample is shown in the Appendix.

In summary the samples are composed primarily of quartz and 'mica' (undifferentiated mica species) with variable amounts of chlorite, K-feldspar, albite, kaolinite, calcite and tourmaline commonly found. More rarely pyrite, cassiterite, gypsum, ?magnetite, fluorite, ?jarosite and ?zaherite are present.

A number of different size and density fractions were analysed (samples 9, 19, and 20) and the intra-samples differences are summarised (Figure 1-3).

Sample 9: There is a reduction in the pyrite and cassiterite concentrations between the –63 µm and light minerals samples (Figure 1).

Sample 19: With the reduction in grain size there is a reduction in the intensity of chlorite and 'mica' peaks and an increase in calcite. Quartz peaks are lowest in the finest fraction (Figure 2).

Sample 20: A similar pattern to sample 19 is observed, with chlorite peaks at their lowest in the smallest size fraction and, to a lesser extent, 'mica' as well. Calcite appears most prominently in the finest grain size with an associated reduction in the intensity of quartz peaks. The mineral zaherite may be identified from the finest size fraction.

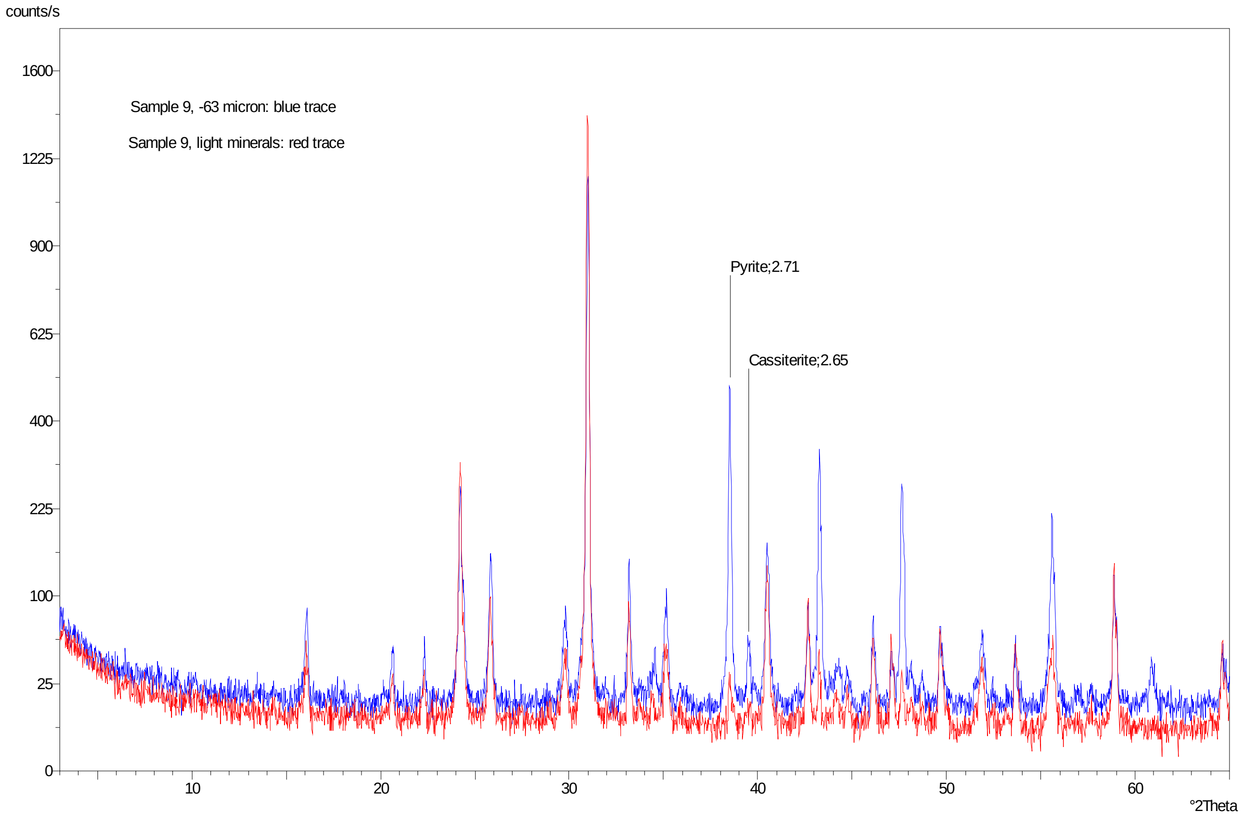


Figure 4: Comparison of the XRD traces for sample 9. Sample '-63 µm' is in red, and 'light minerals' is in blue. X-axis: °2θ Co-Kα; Y-axis: (Intensity) counts per second.

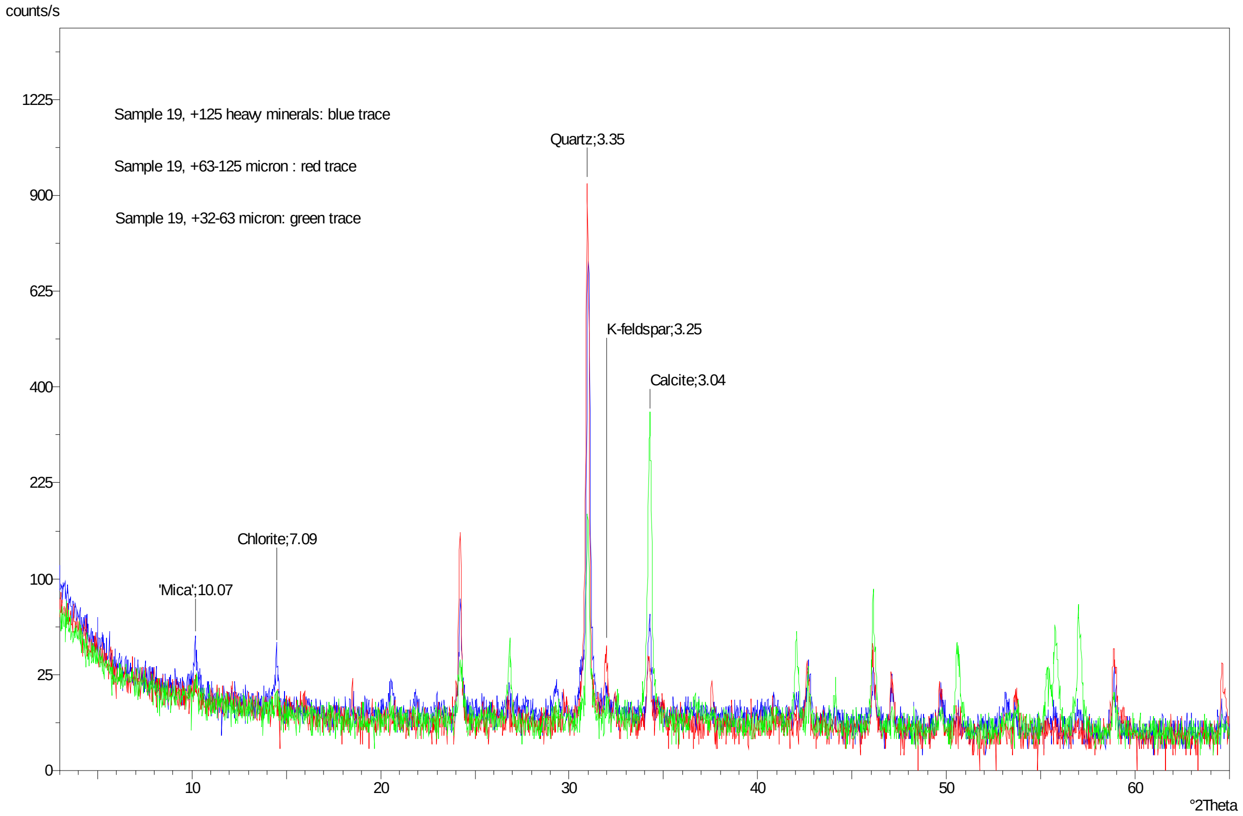


Figure 5: Comparison of the XRD traces for sample 19. Sample '+125 heavy minerals' is in blue, '+63 –125 μm' is in red and '+32 – 63 μm' is in green. X-axis: °2θ Co-Kα; Y-axis: (Intensity) counts per second.

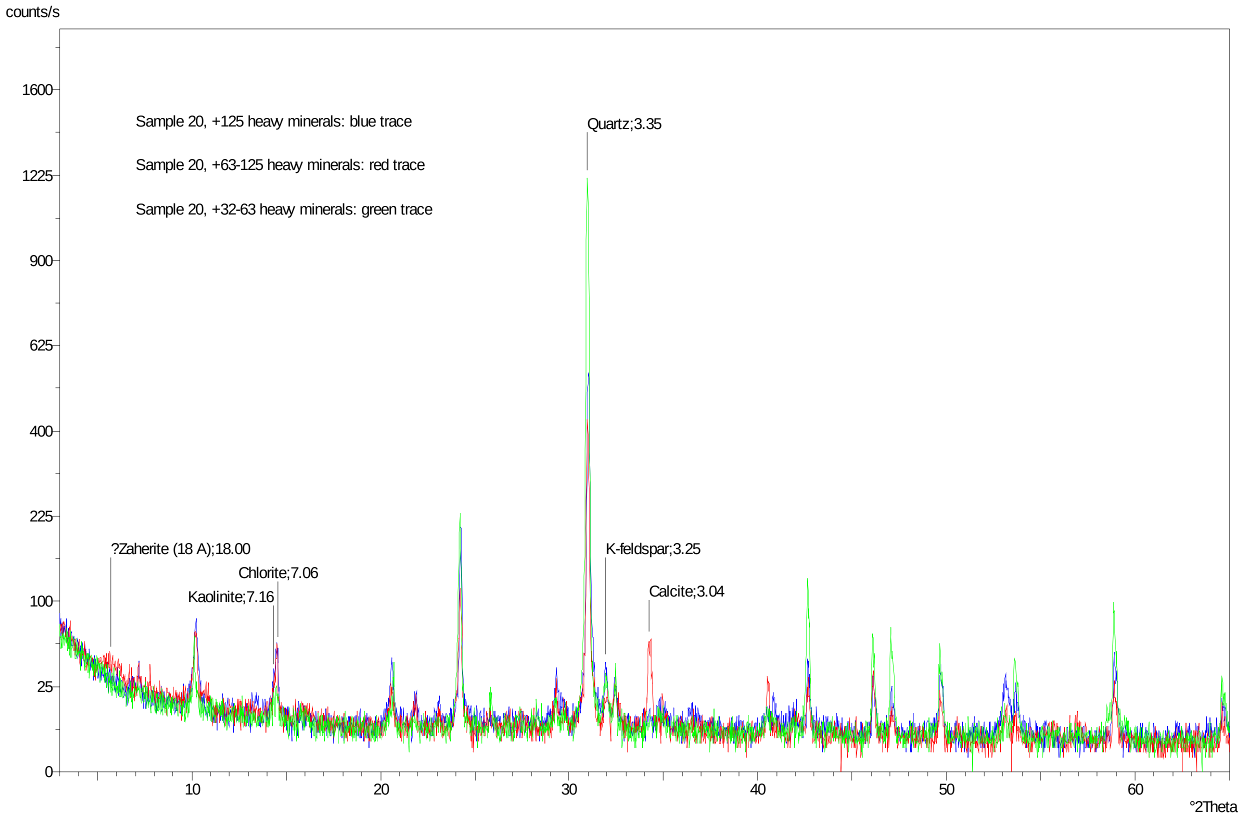


Figure 6: Comparison of the XRD traces for sample 20. Sample '+125 heavy minerals' is in red, '+63-125 heavy minerals' is in blue and '+32-63' is in green. X-axis: °2θ Co-Kα; Y-axis: (Intensity) counts per second.

References

Most of the references listed below are held in the Library of the British Geological Survey at Keyworth, Nottingham. Copies of the references may be purchased from the Library subject to the current copyright legislation.

MCKERVEY, J A. 2002. X-ray diffraction analysis of soil samples from abandoned mines sites in Cornwall. *British Geological Survey Internal Report*, IR/02/137.

Table 2 : Summary of the results of qualitative whole-rock XRD analysis

Sample	MPL code	Mineralogy														
		Quartz	'Mica'	Chlorite	K-feldspar	Albite	Kaolinite	Calcite	Tourmaline	Pyrite	Cassiterite	Gypsum	Magnetite	Fluorite	Jarosiite	Zaherite
9	MPLJ295	mj	tr	-	tr	-	-	-	mi	mj	mi	-	-	-	?tr	-
9	MPLJ296	mj	tr	-	-	-	tr	-	mi	mi	-	-	-	-	?tr	-
14	MPLJ297	mj	tr	mi	mi	mi	tr	mi	mi	-	-	-	-	-	-	-
16	MPLJ298	mj	mi	mi	mi	mi	tr	-	mi	-	-	mi	?tr	-	-	-
18	MPLJ299	mi	mi	mj	-	-	-	-	mi	-	-	-	-	mi	-	-
19	MPLJ300	mj	mi	mi	tr	-	-	-	-	-	-	-	-	-	-	-
19	MPLJ301	mj	tr	-	mi	tr	-	mi	-	-	-	-	-	-	-	-
19	MPLJ302	mi	tr	tr	tr	tr	-	mj	-	-	-	-	-	-	-	-
20	MPLJ303	mj	mi	mi	tr	tr	tr	tr	-	-	-	tr	-	-	-	-
20	MPLJ304	mj	mi	tr	tr	tr	tr	-	-	-	-	-	-	-	-	-
20	MPLJ305	mj	mi	mi	tr	tr	-	mi	-	-	-	-	-	-	-	?tr

'Mica' = undifferentiated mica species

Appendix

Key to the whole-rock X-ray diffraction traces:

X-axis: °2θ Co-Kα

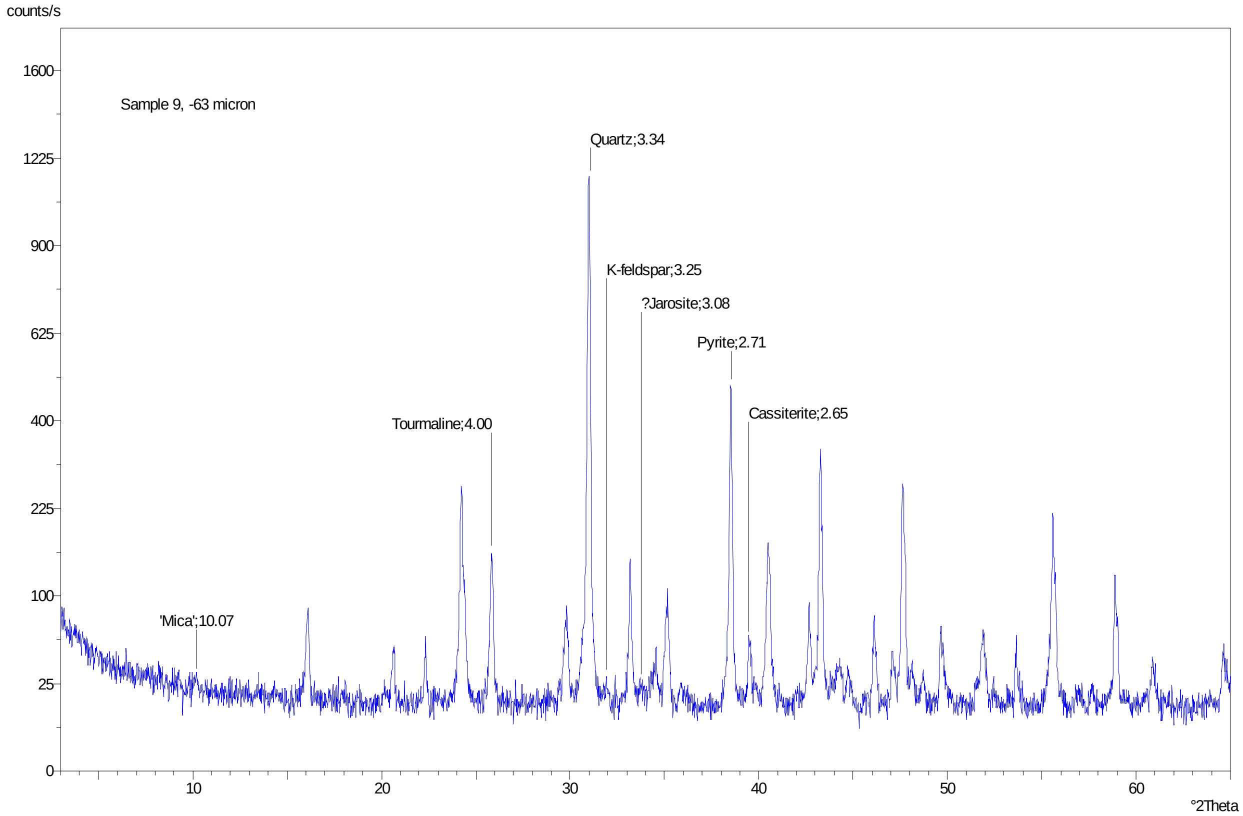
Y-axis: (Intensity) counts per second

Peak Labels: mineral and d-spacing (Å)

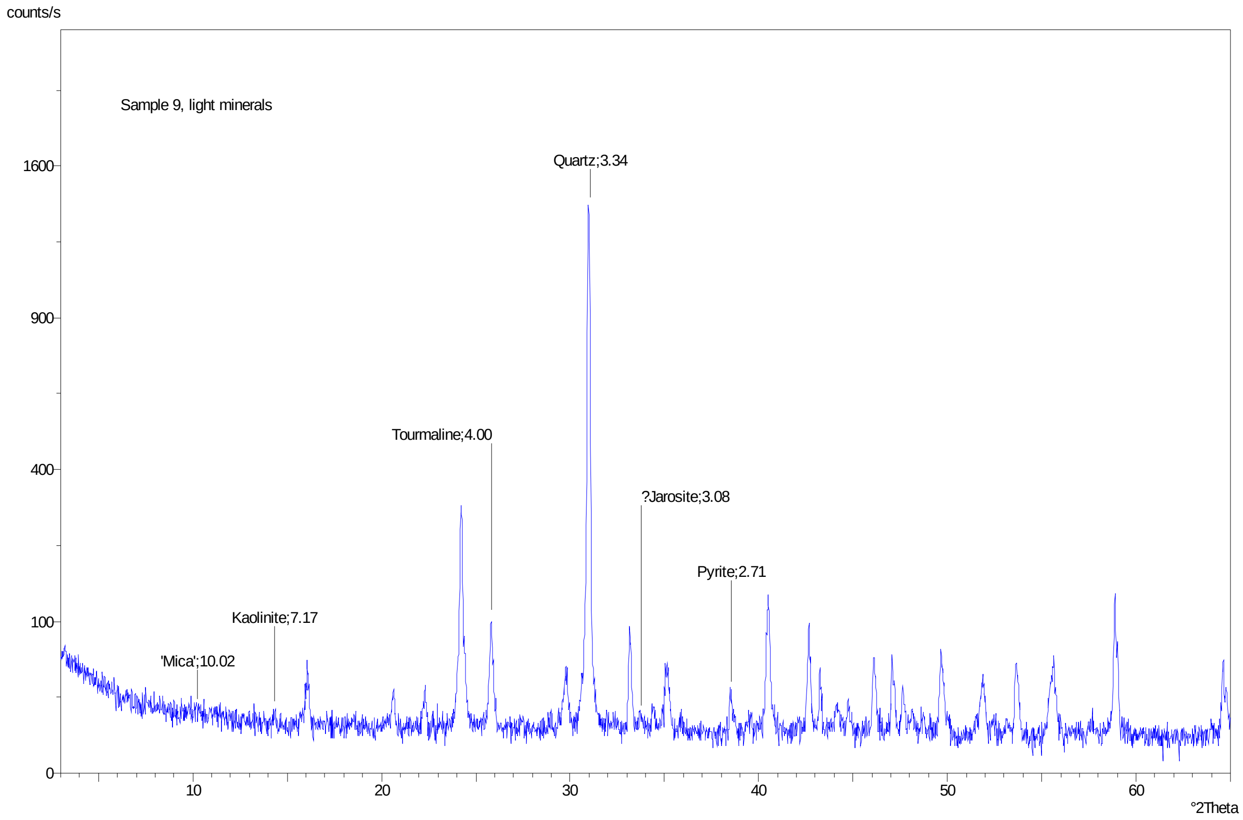
Blue trace: whole-rock powder

Only the most intense/characteristic peak is labelled for each mineral.

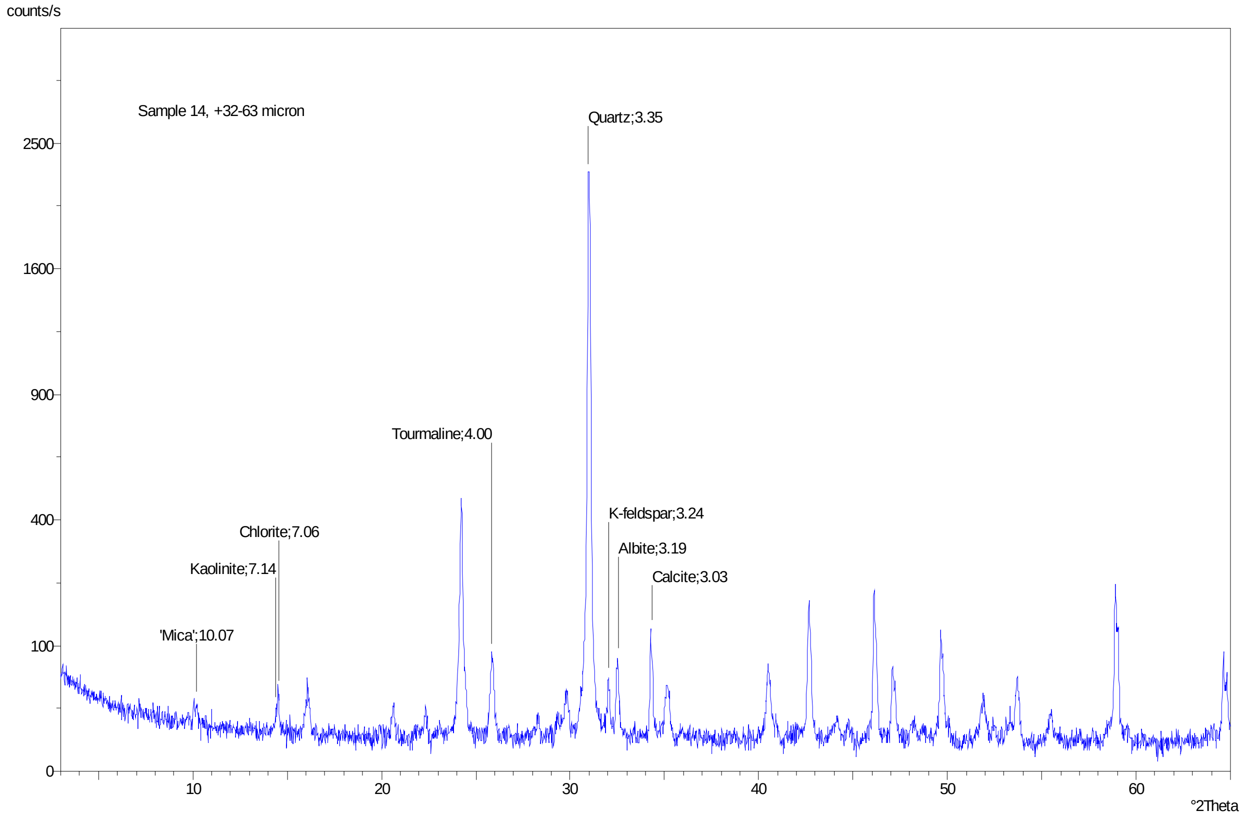
Sample 9, -63 μm



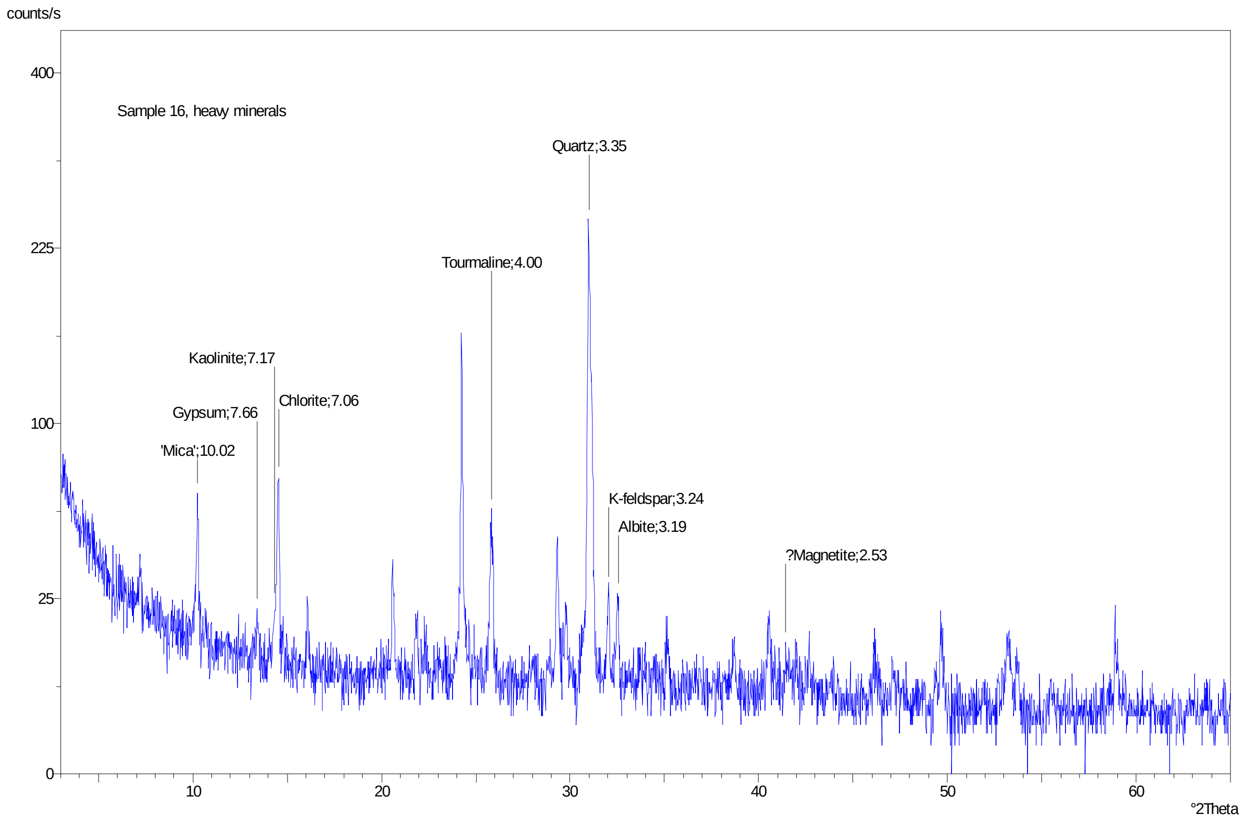
Sample 9, light minerals



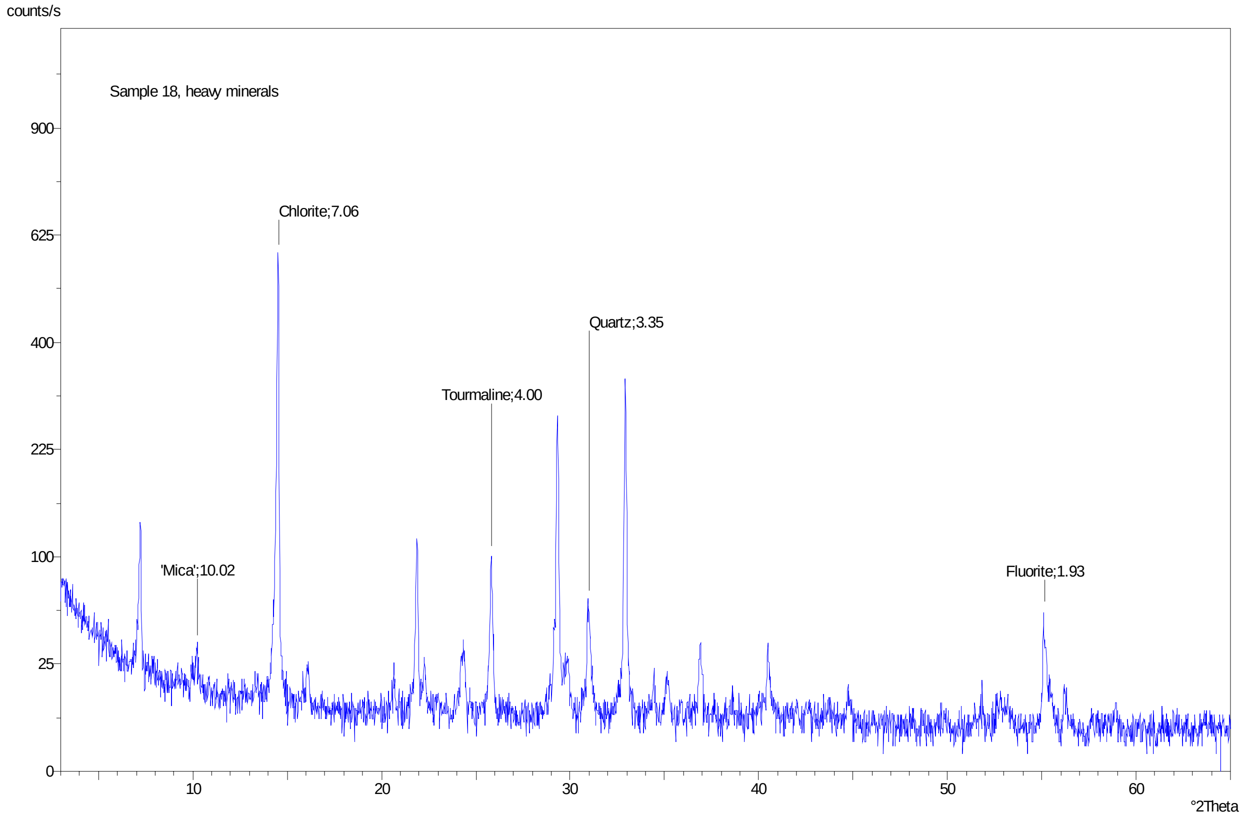
Sample 14, +32 –63 µm



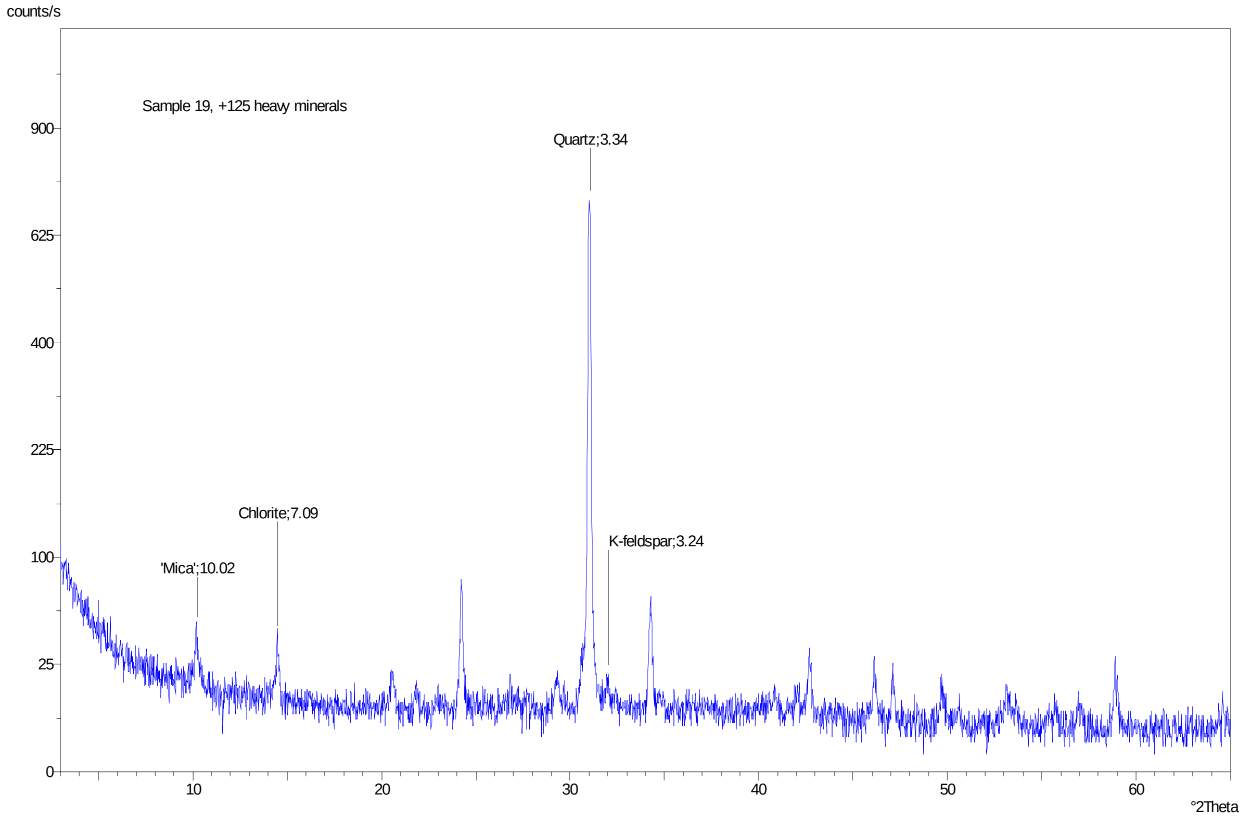
Sample 16, heavy minerals



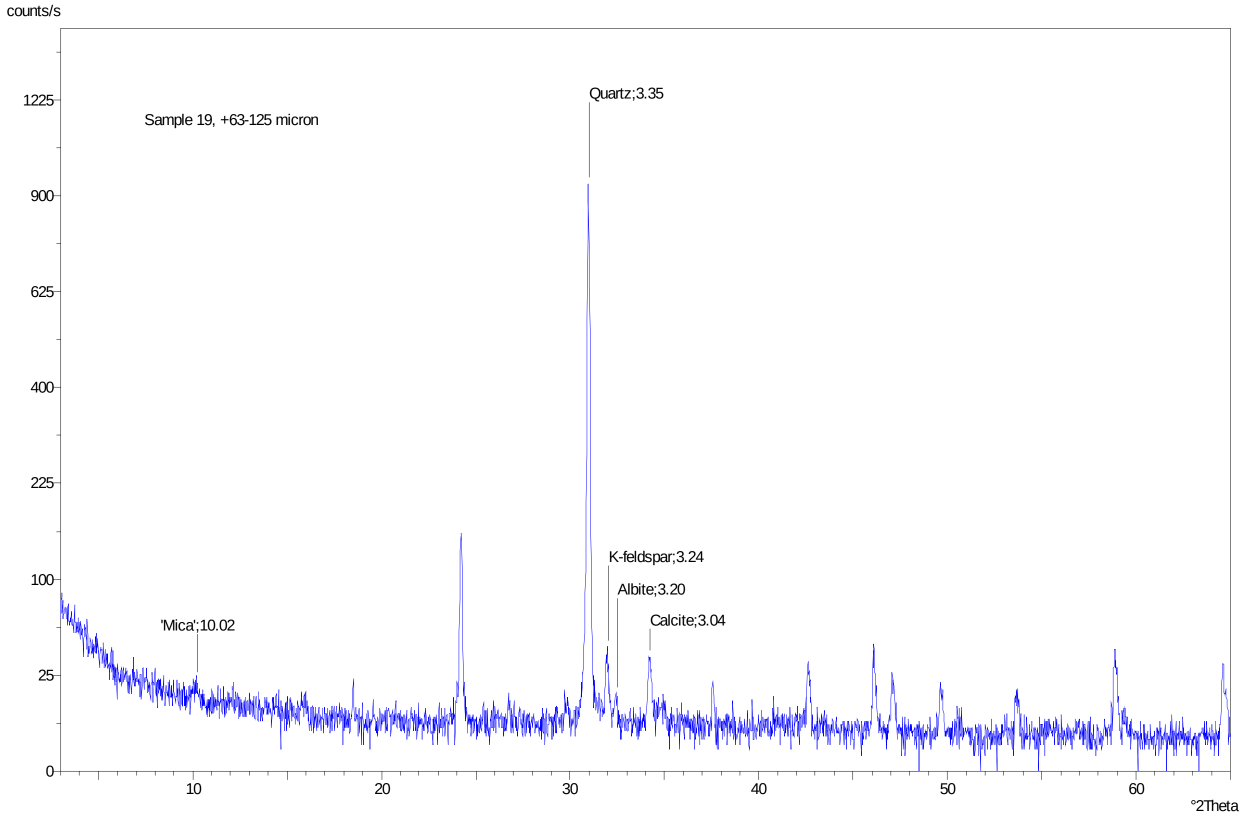
Sample 18, heavy minerals



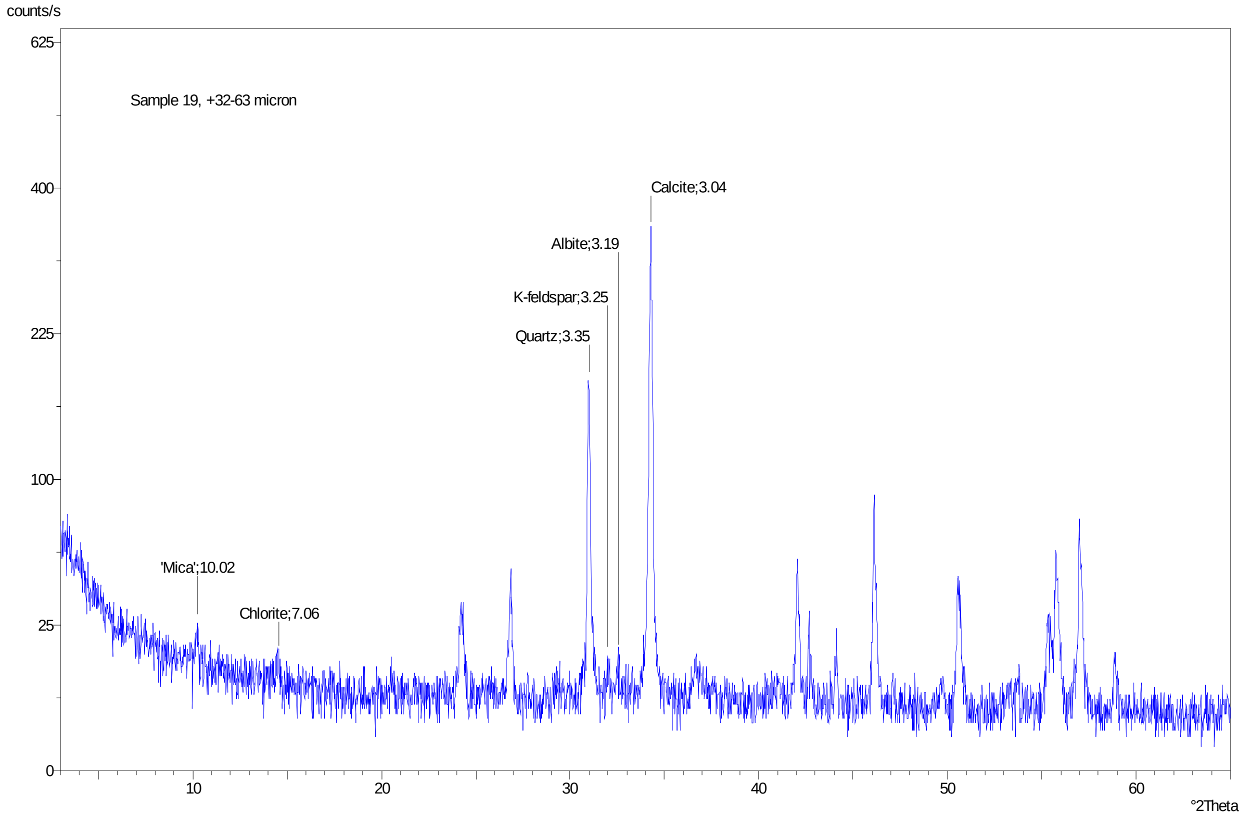
Sample 19, +125 μm heavy minerals



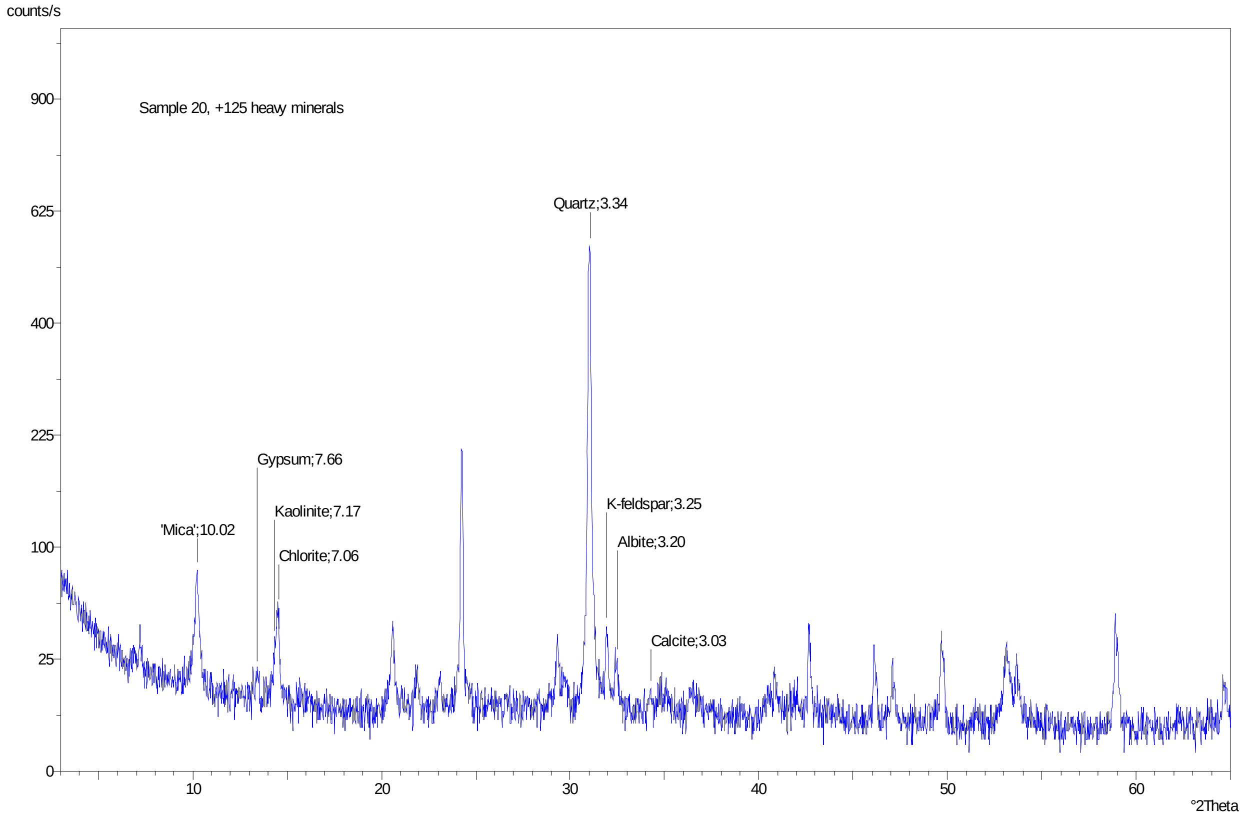
Sample 19, +63-125 μm heavy minerals



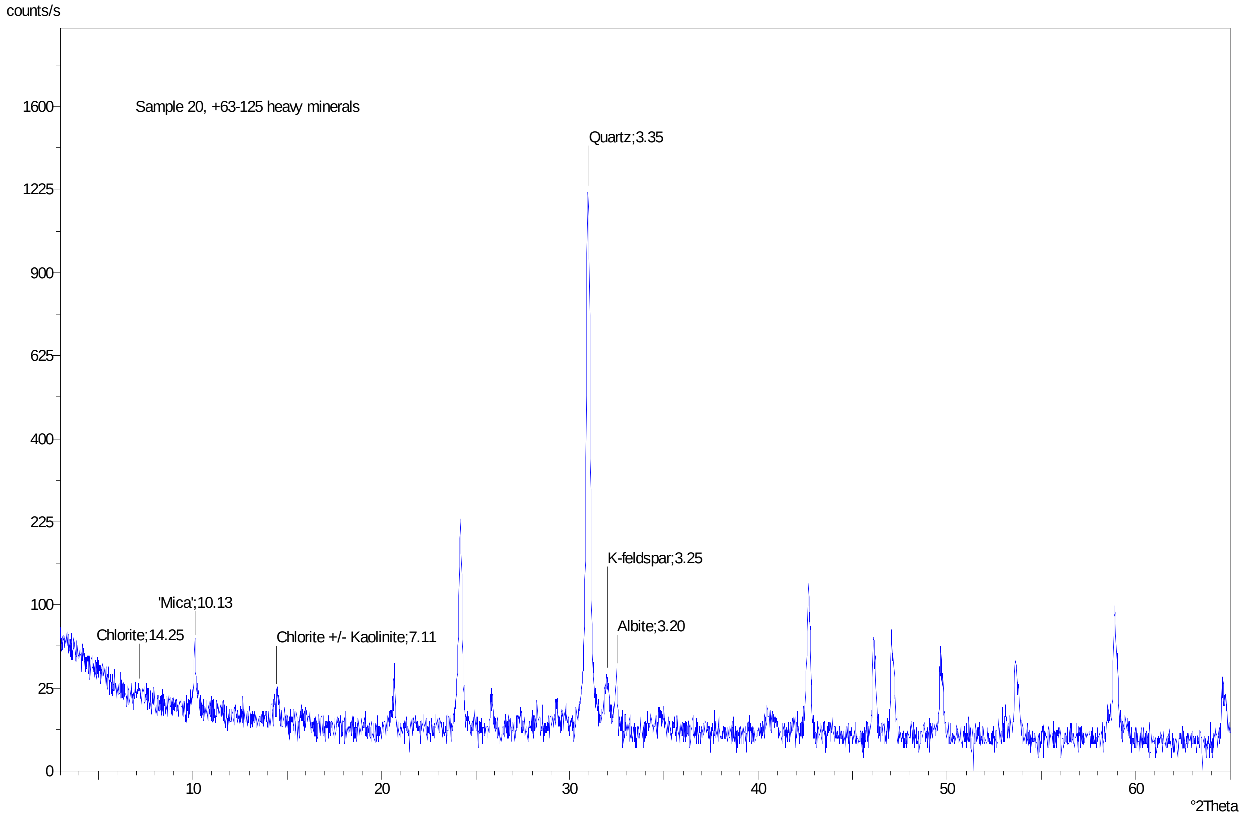
Sample 19, +32-63 μm heavy minerals



Sample 20, +125 μm heavy minerals



Sample 20, +63-125 μm heavy minerals



Sample 20, +32-63 μm heavy minerals