The use of low-toxic heavy suspensions in mineral sands evaluation and zircon fractionation

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Transaction Paper

Synopsis

This paper outlines a simple methodology for mineral characterization, developed as part of the Australian Mineral Industry Research Association (AMIRA) managed research project P777 'The Development of Heavy Suspension Techniques for High Density Separations (Replacement of Clerici's Solution)'. The project was sponsored by De Beers, Rio Tinto and Iluka Resources.

Heavy mineral characterization of samples arising from exploration, mining or metallurgical processes is frequently conducted using laboratory heavy liquid analysis. Unfortunately, there are only a limited number of high density ('heavy') liquids and these tend to be more toxic as their density increases. Low-toxicity inorganic solutions, based on tungsten compounds, have been developed that can be utilized at relative densities (RD) up to 3.0. Beyond this value organic liquids can be used; however, this presents significant health and safety hazards. Diiodomethane (methylene iodide) having a relative density of 3.31 is commonly used. Mixtures of thallium formate and thallium malonate were found in the early 1900s by Clerici to provide liquids having specific gravities between 4.0 and 5.0. For the characterization of the heavy components of mineral sand deposits (e.g. anatase RD 3.9, rutile RD 4.2, ilmenite RD 4.4–4.7 and zircon RD 4.6–4.8) there is currently no heavy liquid alternative to Clerici's solution. Clerici's solution is highly toxic and testing is now conducted by few laboratories worldwide, with costs reflecting the chemical costs, infrastructure costs and health and safety regimes (e.g. blood testing of exposed staff). A simple laboratory technique of density fractionation has been developed, employing suspensions of fine tungsten carbide particles in lithium heteropolytungstates solutions, that can replace Clerici's solution in the evaluation of fine mineral sands samples (e.g. -250 +150 microns). The developing methodology that can achieve low-cost, low-toxic separations at relative densities above 4.0 is outlined and the comparison of results with Clerici's solution presented. In addition, preliminary work on density fractionation of zircon samples is presented. Zircon fractionation relates to their inclusion, radionuclide content and metamictization.

Background

Heavy liquids have wide use in the laboratory for the appraisal of gravity-separation techniques on ores. The aim is to separate the ore samples into a series of fractions according to density, establishing the association between the high and low specific gravity minerals. The mineral grains either 'sink' or 'float' in the heavy liquid selected and are recovered for further analysis.

Organic heavy liquids

Unfortunately, there are only a limited number of high density ('heavy') liquids and these tend to be more toxic as their density increases. The most commonly used heavy liquids in these analyses are volatile halogenated organic solvents (e.g. diiodomethane, relative density 3.31).

Tetrabromoethane (TBE), having a relative density (RD) of 2.96, was commonly used and may be diluted with white spirit or carbon tetrachloride to give a range of densities below 2.96. Using such heavy liquids, acetone can be used as a diluent and for washing the organic from the separated products. Considerable effort must be expected in handling volatile, flammable and toxic organic liquids in sample washing and recovery (recovery of TBE is often only 90%).

Bromoform (relative density 2.89) may be mixed with carbon tetrachloride (relative density 1.58) to give densities in the range 1.58–2.89. For densities of up to 3.3, diiodomethane (methylene iodide) is useful, diluted as required with triethyl orthophosphane. However, this presents significant health and safety hazards.

Mixtures of thallium formate and thallium malonate were found in the early 1900s by Clerici to provide liquids having relative densities up to 4.0 at 20°C, or 5.0 at 90°C, hence 'Clerici's solution' (Wills, 1997). For the characterization of the heavy components of mineral sand deposits (e.g. anatase RD 3.9, rutile RD 4.2, ilmenite RD 4.4–4.7 and zircon RD 4.6–4.8) there is currently no heavy liquid alternative to Clerici's solution. Clerici's solution is highly toxic and testing is now

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conducted by few laboratories worldwide, reflecting the high chemical and infrastructure costs, as well as the strict health and safety regimes required (e.g. blood testing of exposed staff).

Inorganic heavy liquids

Aqueous solutions of sodium metatungstates have certain advantages over organic liquids, such as being virtually non-volatile and non-toxic, and densities of up to 3.1 can be achieved.

A new heavy liquid, lithium heteropolytungstates (LST), was introduced for industrial use at the beginning of 1996 (www.chem.com.au). Specifically developed as a safe and effective replacement for bromoform and TBE, LST has low viscosity and high thermal stability with an expected operating density of 2.85 g/ml.

These low-toxicity inorganic solutions, based on tungsten compounds, can be utilized at relative densities (RD) up to 3.0. However, beyond this value currently only organic liquids can be used.

Heavy suspensions

In order to achieve separation densities above 3.0 heavy suspensions can be used for float/sink separations.

'Cargille' liquids, heavy metal particles dispersed in organic liquids, have been produced with relative densities ranging up to 7.5 (Browning, 1961). The use of these liquids was limited to separation of coarser particle sizes, usually larger than 0.6 mm, due to the suspension physical properties. The heavy metal particles in Cargille liquids settle slowly and form a soft mass at the bottom of the suspension. Before use, the suspension must be stirred to disperse the metal particles uniformly throughout the liquid. An alternative is the use of mercury-bromoform emulsions (after Burt and Mills, 1984), which have a maximum relative density of 7.0, and can be used successfully on particles as small as 0.1 mm.

Overbeek (1986) has utilized tetrabromoethane (1,1,2,2-tetrabromoethane) with the addition of ferrosilicon in order to obtain relative densities in the range 3.1 to 4.1.

Rhodes, Miles and Hall (1993) have developed a technique using finely divided ferrosilicon in solutions of sodium polytungstate (SPT) for high-density separations. The use of heavy suspensions, comprised of lithium heteropolytungstates (LST) with ferrosilicon, for sink-float analysis has been demonstrated by Eroglu and Stallknecht (2000). Both of these studies used relatively coarse mineral particle sizes (+1 mm).

Mineral sand mineralogy

Mineral sand resources almost always contain more than one valuable (and relatively heavy) mineral. Titanium minerals are found with a large range of titanium contents, giving rise to density variation and often subjective mineralogical descriptions. Zircon, the other major mineral sands product, provides its own challenges during resource assessment and metallurgical test work (Gilman and Hugo, 2003). Companies tend to rely on laboratory heavy liquid separation in the evaluation of samples arising from exploration, mining or metallurgical processes.

The natural alteration of ilmenite by the partial removal of iron results in an intermediate iron titanate of poorly defined structure for which the name 'pseudorutile' has been proposed. Complete removal of iron from the pseudorutile lattice results in a grain composed of crystallites of the minerals rutile and anatase. The term 'leucoxene' is applied to the high-TiO_2 products of alteration. Various compositional ranges (or minimum contents) of TiO_2 are used within the industry for naming the various minerals (see Table I).

Anatase is a polymorph with rutile (i.e. both have the same chemistry as TiO_2, but they have different structures). From a mineralogical perspective, the alteration of ilmenite to rutile or anatase creates a range of mineral phases, which may be found as discrete grains, or more commonly as intergrowths within grains (Gilman and Hugo, 2003). The range of compositions of leucoxene will give rise to a spectrum of particle densities, the higher the iron oxide content the higher the density.

Zircon (relative density 3.9–4.8) is the classic example of a mineral that undergoes the process of metamictization (Holland and Gottfried, 1955). Metamictization is a natural process of radioactive decay resulting in gradual and ultimately complete destruction of a mineral's crystal lattice, leaving the mineral amorphous. Holland and Gottfried demonstrated that the effect could reduce zircon relative density from 4.7 for 'pristine' zircon progressively to values around 3.95. Unaffected specimens are sometimes termed high zircon while metamict specimens are termed low zircon.

The majority of commercially exploited mineral sand deposits contain the valuable heavy mineral grains in the size range 100–250 microns; however, a number of significant deposits are known (particularly in the Murray Basin region of Australia) that contain finer (50–100 micron) grains.

Methodology

Mineral sands types and sizes

A 'rutile' sample was received from one of the AMIRA project sponsors. The sample sizing substantially -250 +90 micron and was sieved at –250+150 micron and –150+90 micron for test work.

Samples of cleaned 'ilmenite' concentrates were also supplied by the project sponsors, allowing both -250+150 micron and -150+90 micron size fractions to be sieved for subsequent test work.

Zircon was obtained from project sponsors and has been sieved into two fractions: -250+150 micron and –150+90 micron and Western Australian zircon concentrate sample (-250+125 μm).

Table I

Simplified classification of TiO_2-containing minerals

Heavy mineral name	Specific gravity range	Formula	TiO_2 content
Ilmenite	4.7–4.79	$FeTiO_3$	35–65
Pseudorutile	3.9	TiO_2	60–65
Leucoxene	4.2–3.9	$FeTiO_3 – TiO_2$	65–90+
Anatase	3.8–3.9	TiO_2	>90
Rutile	4.2–4.3	TiO_2	94–96 (commercial close to 100)

The use of low-toxic heavy suspensions in mineral sands evaluation and zircon

A kyanite sample was received from one of the AMIRA project sponsors. The sample sizing was substantially in the range -250 +150 micron and was sieved at these sizes for test work.

Chemicals

Lithium heteropolytungstates (LST) was supplied by Central Chemical Consulting, Perth, Western Australia (www.chem.com.au) with a density 2.85 g/ml and viscosity about 11 cP. Solutions of a specific density were prepared by diluting with deionized water or by concentrating by evaporation.

Various tungsten carbide powders were used to generate heavy suspensions; however, the majority of test work was conducted using a nominal size of 3.3 micron, density 14.85 g/cm^3 (Table II).

Density measurements

Precise determination of the density of suspension is important for accurate sink/float separation. A 50 ml volumetric flask was used to weigh a known volume of LST heavy liquid. Density measurements of suspensions were carried out by weighing a known volume of suspension using a 1 000 ml cylinder and an FA-2000 electronic balance. Between measurements and tests, the LST and the suspension samples were covered to prevent evaporation.

Density measurements of mineral sands components and sink/float fractions were determined by using a Pycnometer method as Australian Standards 1141.6.2 (1996).

Determination of mineralogical composition of samples

Float and sink fractions were analysed by optical mineralogy and X-ray diffraction (XRD). Samples were ground in a zirconia ring mill for 3 minutes before loading sideways into XRD cavity mounts. XRD conditions were Siemens D500 X-ray diffractometer, Ni filtered-Cu Kα radiation 40 kV/30 mA, scan rate 1°2θ/min, 0.02° step from 3° to 81°2θ, 1°/1° div and 0.15 mm rec slits. Mineral phases were identified from the digitized traces by computer aided search/matches of the 2004 ICDD PDF-4 mineral sub-file. Quantitative mineralogy has been determined using SiroQuant™ version 3 software package. The results should be considered semi-quantitative.

Float/sink tests with heavy suspensions

Suspensions of tungsten carbide (14.85 relative density) and LST (2.85 relative density) are made up in a 250 ml beaker. A weighed quantity of tungsten carbide is added to the required volume of LST to obtain the suspension density required in each test condition (refer Table III).

A float/sink test was carried out on a 6 g mineral sample with size fraction –250+150 μm or –150+90 μm using 180–230 ml suspensions of LST and tungsten carbide of progressively increasing density (SG 3.45, 3.55, 3.65, 3.75, 3.85, 3.95, 4.05, 4.15, 4.25, 4.35, 4.45, 4.55 and 4.65). The float and sink fraction of the mineral sand in each suspension was collected, washed with deionized water (250 ml) and oven dried to constant weight (at 105°C) before being weighed. This procedure was repeated on a new 6 g sample.

The suspension is agitated for a period of 2 minutes to thoroughly wet and disperse the tungsten carbide particles. The mineral sample to be tested is then added to the beaker and intensely stirred (500 rpm) for 2 minutes.

After two minutes, stirring is halted and the suspension is quickly decanted to a modified settling funnel (Figure 1). The 250 ml separating funnel has been modified by removing the valve ('tap') and fixing a piece of flexible polymer tubing

Table II

Particle size of Sandvik tungsten carbide powders (microns)

Grade	Grain size (Sandvik)	Comment
WC100	1.1 ± 0.5	Fine
WC300	3.3 ± 0.8	Medium
WC500	5.3 ± 0.8	Medium coarse
WC1000	10.5 ± 1.5	Coarse
WC1500	15 ± 3.0	Extra coarse

Table III

Potential tungsten carbide—LDT suspension compositions (RD LST = 2.85)

Suspension relative densities from	% Tungsten carbide by volume	% Tungsten carbide by weight
3.45	5.0	21.5
3.55	5.8	24.3
3.65	6.7	27.2
3.75	7.5	29.7
3.85	8.4	32.2
3.95	9.2	34.6
4.05	10	36.7
4.15	10.9	38.8
4.25	11.7	40.7
4.35	12.5	42.6
4.45	13.4	44.5
4.55	14.2	46.2
4.65	15	47.9
4.75	15.9	49.5

Figure 1—Modified separating funnel

to the base of the separating funnel. The polymer tubing is sealed with a pinch clip, which is opened to release the sinks fraction at completion of each experiment. In most experimentation a 'separation time'—the time for 'sinks' and 'floats' fractions to separate—of 10 minutes is used. It should be noted that during this time the tungsten carbide also settles in the LST, creating a higher density of separation than that of the initial suspension. A small depth (2–3 mm) of clear LST is typically observed on top of the suspension. The dark opaque suspension makes it difficult to observe the float or sink fractions of the mineral samples.

At the end of the separation time, one-third of the total volume is removed through a clean sieve as containing the 'sinks' fraction (Figure 2), unlike the 90% recommended in AS4350.2 (1999). The pinch clip is again closed prior to the release of the remaining suspension, including the floats fraction, through a clean sieve to retain the floats sample.

The recovered suspension passing through each sieve is collected for reuse. The sinks and float fractions are washed with deionized water through a sieve with an aperture smaller than the particle size of the mineral sand. The washed float and sink samples are oven dried at 105°C until a stable weight is achieved and the sample is weighed.

The 'washings' that pass through the sieve are retained for the recovery of tungsten carbide by filtration and LST by evaporation to the initial solution density.

Results

Float/sink testing

A 6 g sample of zircon and kyanite (50/50 by weight) with particle size –250+150 micron was prepared and a float/sink analysis conducted in order to confirm that a suspension method could separate these minerals.

The densities of the zircon and kyanite samples were determined by a pycnometer. The zircon sample average relative density was 4.6 and kyanite 3.6. It was decided to choose an initial density of suspension at which all zircon should report to a sink fraction and all kyanite should be recovered as a float fraction. The minerals were separated at 3.75 g/ml initial density of suspension. The results of the test showed that 3.04 g (50.7%) of the sample reported to floats and 2.95 g (49.2%) of the sample reported to sinks. Densities of the floats and the sinks were determined by pycnometer (Table IV).

The test results show full separation of kyanite and zircon, which prove that suspensions could be employed for sink/float separation of minerals having relative densities above 3.1.

Individual mineral testing

Individual mineral samples (rutile, ilmenite, zircon) with size fraction –150+90 micron have been tested across a range of suspension densities.

The rutile sample relative density was determined using a pycnometer method (Australian Standard AS 1141 Method 6.2: Particle density and water absorption of coarse aggregate—pycnometer method) as averaging 4.13.

The ilmenite sample relative density was determined using the same pycnometer method as averaging 4.35.

The zircon sample relative density was determined using the same pycnometer method as averaging 4.63. The kyanite sample relative density was determined using the same pycnometer method as having an average value of 3.65.

The per cent by weight of mineral recovered to float in each density range from separations conducted using tungsten carbide/LST suspension is shown below in Table V for –150+90 micron rutile, ilmenite and zircon. In all tests 6 grammes of mineral sample is used.

Figure 2—Removing 'sinks' fraction through a clean sieve

Table IV

Pycnometer density determinations

Sample	Relative density
Kyanite	3.649
Zircon	4.604
Zircon/kyanite (50/50 wt) mix (feed)	4.105
Zircon/kyanite (50/50 wt) mix (sinks)	4.598
Zircon/kyanite (50/50 wt) mix (floats)	3.644

Table V

Sink/float analysis of –150+90 μm single phase (density of LST 2.85 g/ml)

Initial suspension relative density	Rutile	Ilmenite	Zircon
RD 3.45 floats (wt%)	6.3	4.0	0.4
RD 3.55 floats (wt%)	23.5	2.6	
RD 3.65 floats (wt%)	48.9	37.3	7.4
RD 3.75 floats (wt%)	68.8	34.5	
RD 3.85 floats (wt%)	71.0	45.6	46.0
RD 3.95 floats (wt%)	78.0	58.7	58.9
RD 4.05 floats (wt%)	85.1	72.4	
RD 4.15 floats (wt%)	84.5	80.6	61.1
RD 4.25 floats (wt%)	85.8	85.2	71.6
RD 4.35 floats (wt%)	88.3	91.3	75.6
RD 4.45 floats (wt%)	91.3	90.3	83.6
RD 4.55 floats (wt%)	90.4		
RD 4.65 floats (wt%)	84.3		

- For fraction size –150+90 micron around 91% of the mass of a 'rutile' sample of average relative density of 4.13 has been recovered to a float fraction using an initial suspension relative density of 4.45
- Around 91% of the 'ilmenite' sample (–150+90 micron) can be floated at an initial suspension relative density of 4.35
- Using an initial suspension relative density 4.65, approximately 84% of –150+90 micron zircon could be recovered as a float fraction.

The results are shown graphically in Figure 3.

Experimental reproducibility

The results of duplicate experimentation on the -250+150 micron rutile sample are shown in Table VI; fraction masses are in grammes.

The results of duplicate experimentation on the -250+150 micron kyanite sample are shown in Table VII, fraction masses are in grammes.

A sample of zircon and kyanite (50/50 by weight) with particle size -250+150 micron was tested for experimental reproducibility (Table VIII; fraction masses are in grammes).

XRD and pycnometer analysis of float-sink fraction of rutile

Selected floats and sinks fractions from rutile separation have been analysed using semi-quantitative X-ray diffraction (XRD) for their mineralogical composition (Table IX) and pycnometer (density bottle) determinations for their average relative density.

Table X shows the pycnometer density determinations. These results confirm the effective density separation of the 'rutile' sample.

The relative density of anatase is 3.8–3.9, whereas rutile has a relative density of 4.2–4.3. Applying a weighted average to the composition of the sample indicated by XRD is in approximate agreement with these values, though it requires a 'rutile' relative density of nearer to 4.15 if the sample is a simple binary mixture of two phases of exact relative densities.

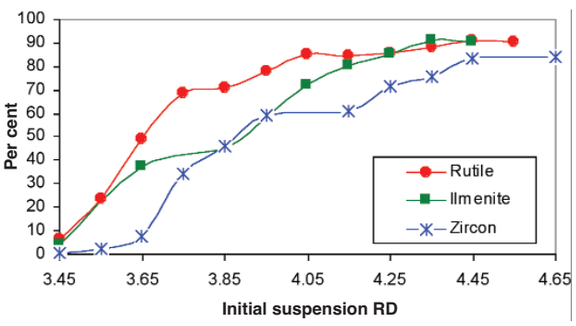

Figure 3—Float/sink analysis of a –150+90 μm rutile, ilmenite, and zircon sample in heavy suspensions showing percentage of sample reporting to 'float' fraction versus initial suspension relative density

Table VI

Results of sink-float testing on 6-gramme sample of –250+150 μm 'rutile' sample

Fraction	Floats	Sinks
Initial suspension RD 3.5	0.77 ± 0.10	5.09 ± 0.05
Initial suspension RD 3.75	2.10 ± 0.08	3.86 ± 0.11
Initial suspension RD 3.85	2.98 ± 0.11	2.86 ± 0.02
Initial suspension RD 3.95	5.45 ± 0.08	0.64 ± 0.06
Initial suspension RD 4.05	5.76 ± 0.02	0.29 ± 0.02

Table VII

Results of sink-float testing on 6-gramme sample of –250+150 μm kyanite sample

Fraction	Floats	Sinks
Initial suspension RD 3.25	0.09 ± 0.01	5.91 ± 0.01
Initial suspension RD 3.35	0.37 ± 0.08	5.61 ± 0.08
Initial suspension RD 3.45	4.86 ± 0.01	1.14 ± 0.01
Initial suspension RD 3.55	5.51 ± 0.01	0.50 ± 0.01
Initial suspension RD 3.65	5.84 ± 0.00	0.14 ± 0.01
Initial suspension RD 3.75	5.93 ± 0.01	0.07 ± 0.00

Table VIII

Results of sink-float testing on 6-gramme sample of –250+150 μm zircon/kyanite (50/50 by weight) sample

Fraction	Floats	Sinks
Initial suspension RD 3.75	3.04 ± 0.02	2.95 ± 0.03

Table IX

Semi-qualitative XRD analysis of rutile feed and selected sink-float fractions (wt%)

	Anatase	Rutile	Tungsten carbide	Quartz
Feed	5.2	94.7	0.1	n.a.
3.5 float	18.9	76.9	0.1	4.2
3.5 sink	6.6	91.3	0.3	1.4
3.75 float	11.6	87.5	0.9	n.a.
3.75 sink	0.6	87.4	12.0*	n.a.
3.85 float	10.9	86.3	0.1	2.8
3.85 sink	0.4	98.4	0.1	1.1

* indicates significant tungsten carbide contamination in early testing; n.a. indicates not analysed

Table X

Pycnometer density determinations of –150+90 μm ilmenite

	Feed	3.85	3.95
SG—float	4.35	4.0262	4.1978
SG—sink	4.3381	4.4274	

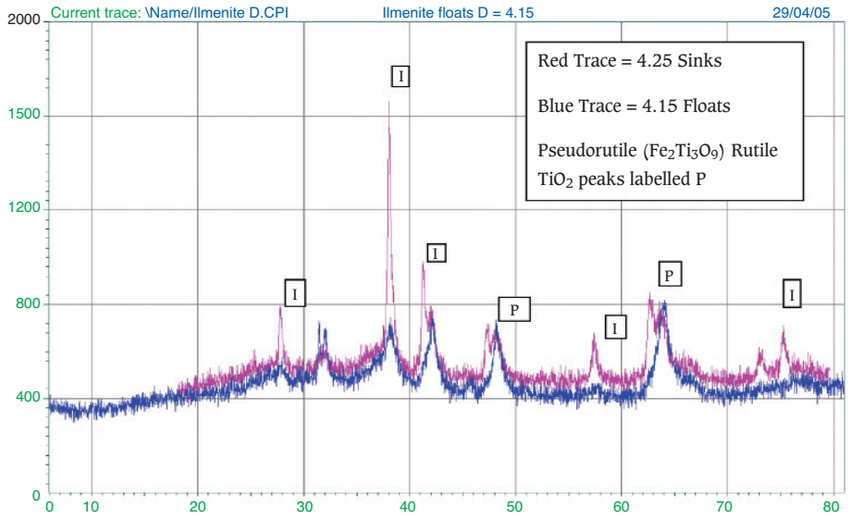

Figure 4—XRD traces from RD 4.25 sinks of 'ilmenite' sample and RD 4.15 floats of 'ilmenite' sample

XRD and optical microscopic analysis of float-sink fraction of ilmenite

The ilmenite density fractions differ slightly in colour and give dramatically different XRD traces, indicative of low-Ti and high-Ti mineral compositions.

X-ray diffraction analysis of the floats fraction at the initial suspension relative density of 4.15 shows the fraction to be comprised of pseudorutile ($Fe_2Ti_3O_9$) and rutile, whereas the 4.25 sinks fraction is ilmenite (Figure 4). This indicates the density fractionation of low-Ti from high-Ti 'ilmenite'.

XRD and optical microscopic analysis of float-sink fraction of zircon

A significant difference in the microscopic appearance of the zircon fractions was observed under optical microscopy. Under constant lighting conditions the floats fraction at 4.25 initial suspension relative density are predominantly 'cloudy' opaque grains with the sinks fraction at 4.45 initial suspension relative density composed of bright clear transparent grains (Figures 5 and 6). The process of metamictization can explain this.

Table XI shows the pycnometer density determinations. These results confirm the effective density separation of the zircon sample.

Metamict zircon: an analysis after sink-float separations

A Western Australian zircon concentrate sample (-250+125 μm) was separated into low, medium and high density fractions. The separations were performed at densities SG 4.00 and 4.25.

After the sample was separated, dried and weighed, fractions were analysed by stereo and pol-microscopy. Images were obtained from polished sections produced from fractions using backscattered electron imaging.

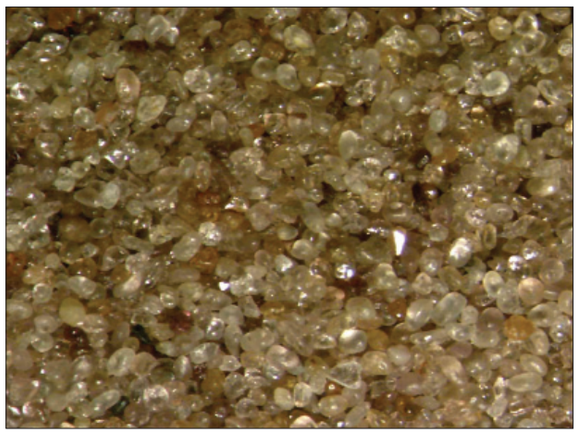

Figure 5—The float fraction of –250+150 μm zircon at 4.25 initial separation relative density

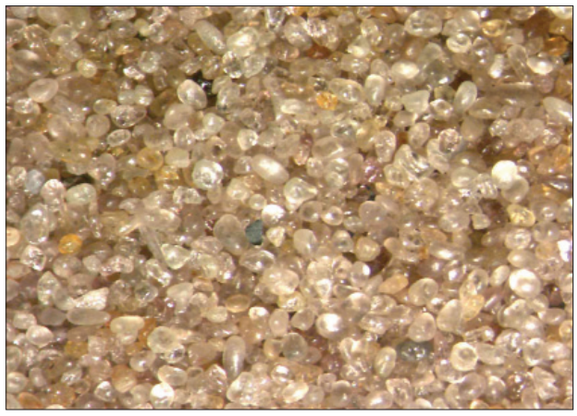

Figure 6—The sink fraction of –250+150 μm zircon at 4.45 initial separation relative density

Table XI

Pycnometer density determinations of –150+90 µm zircon

	Feed	3.85	3.95
SG—float	4.5962	4.5838	4.5882
SG—sink	4.6547	4.6617	

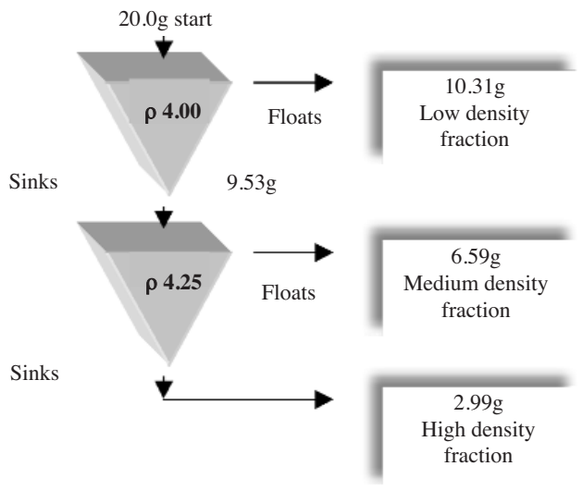

Figure 7—Diagram of the Western Australian zircon concentrate separation

The procedure for fractionation is shown in Figure 7.

Stereo-microscopic images

As the zircon density increases after the sink-float separations it can be seen from the stereo-microscopic images (Figures 8–11) that the grains become clearer and more transparent.

Holland and Gottfried (1955) demonstrated that the SG of zircon decreases with increased metamictization. Therefore it is suggested that this zircon sample has undergone more radioactive decay because of either the age of the sample, or because; it contains more actinides than the other zircon samples tested, which have resulted in significant alteration of its crystal lattice and therefore decreased its density.

Pol-microscopic images

The grains from this Western Australian zircon product tend to be fractured and many grains in the low density fraction show an orange/brown coloration due to inclusions (Figures 12 and 13) while some grains display a pink hue.

Results of comparison of composite sample sequential suspension testing with Clerici's solution

A 90-gramme sample of –250 +150 micron rutile-ilmenite-zircon (33% of each) was prepared and sent to a commercial testing laboratory in order to compare heavy suspension performance against Clerici's solution. The laboratory conducted float/sink tests with the sample over 6 relative densities at 0.1 increments of Clerici's solution between 3.85 and 4.35. The 4.35 RD solution is the limit of commercially available testing via this laboratory.

Similar mass fractionation to that achieved in the commercial laboratory was obtained utilizing the heavy suspension methodology. An additional benefit of this methodology was being able to fractionate at relative

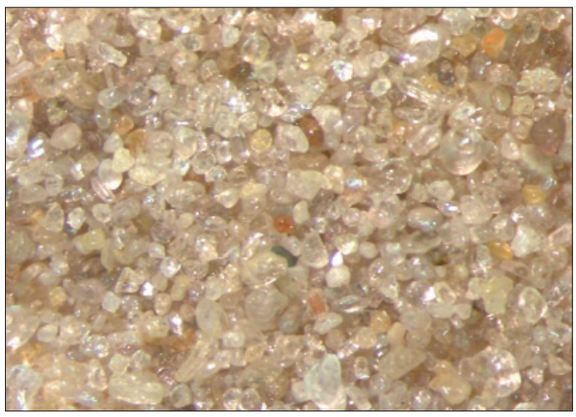

Figure 8—Western Australian zircon concentrate

Figure 9—Low density zircon fraction

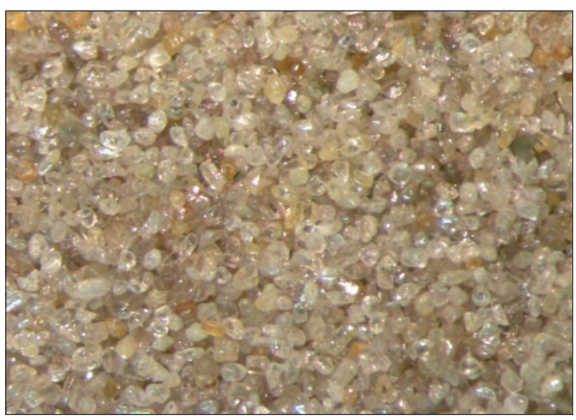

Figure 10—Medium density zircon fraction

densities above 4.35 (i.e. 'cut' into the zircon content). The results obtained from the testing laboratory and a sequential heavy suspension method are given in Table XII.

Semi-quantitative X-ray diffraction of the density fractions has confirmed similar mineralogical content in the Clerici's solution fractions to that of the heavy suspension density fractionation (Table XIII; Figures 14 and 15).

Figure 11—High density zircon fraction

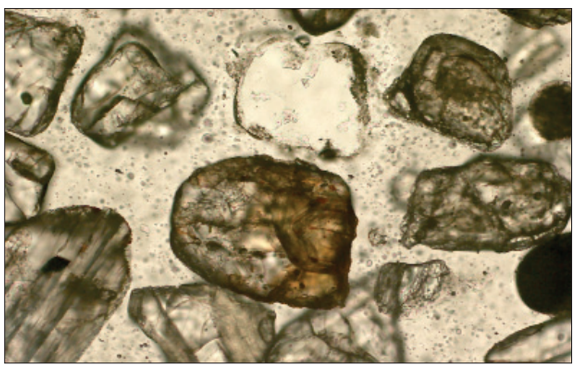

Figure 12—Low density zircon fraction, x 20

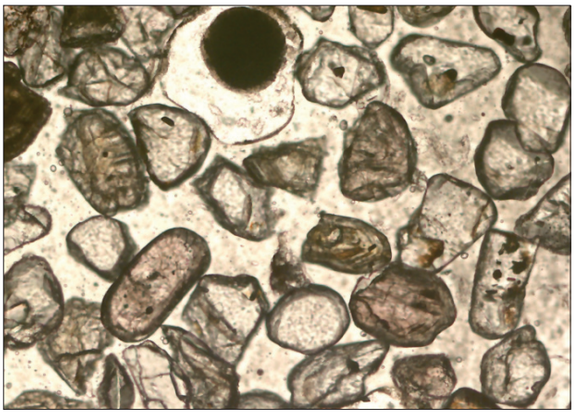

Figure 13—Low density zircon fraction, x 10

Table XII

Density fractionation results from Clerici's solution and heavy suspension characterization of –250+ 150 μm rutile-ilmenite-zircon samples

Solution RD or initial suspension RD	Incremental 'floats' mass wt% Clerici's solution	Incremental 'floats' mass heavy suspension wt%
3.45	-	6.76
3.55	-	6.12
3.65	-	9.51
3.75	-	14.69
3.85	3.30	14.49
3.95	3.37	13.78
4.05	5.71	12.97
4.15	5.13	9.31
4.25	23.16	6.92
4.35	4.50	3.53
4.35 'Sinks'	54.45	-
4.45	-	1.45
4.45 'Sinks'	-	0.47
Total	100	100

Table XIII

Results of semi-qualitative XRD analysis of selected float fractions from Clerici's solution and heavy suspension tests (wt%)

Solution RD or initial suspension RD	Anatase		Ilmenite		Rutile		Zircon		Quartz	
	HS	Clerici	HS	Clerici	HS	Clerici	HS	Clerici	HS	Clerici
3.45	15.5	-	1.3	-	78.2	-	0.6	-	4.4	-
3.55	15.8	-	2.6	-	66.1	-	15.2	-	0.3	-
3.65	9.0	-	3.8	-	81.7	-	5.3	-	0.3	-
3.75	1.3	-	3.7	-	91.0	-	3.8	-	0.2	-
3.85	0.0	29.9	6.0	2.5	70.9	59.1	23.0	0.3	0.0	8.1
3.95	1.9	28.7	6.7	2.4	55.2	66.4	35.6	1.0	0.6	1.5
4.05	0.3	3.4	5.5	2.0	20.9	90.3	73.3	2.0	0.0	2.2
4.15	0.9	0.2	5.2	2.5	7.5	96.1	86.3	1.0	0.1	0.2
4.25	0.0	0.0	0.0	1.2	2.4	97.4	97.4	0.9	0.2	0.5
4.35	0.5	0.0	0.1	2.6	2.6	97.4	96.6	00	0.2	0.0
4.35 sinks	-	0.5	-	0.0	-	3.8	-	84.8	-	0.5
4.45	0.4	-	0.1	-	1	-	98.3	-	0.2	-

The use of low-toxic heavy suspensions in mineral sands evaluation and zircon

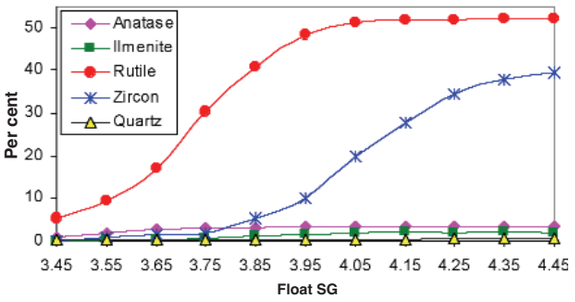

Figure 14—Results of semi-qualitative XRD analysis of accumulative float fractions from heavy suspension tests (wt%)

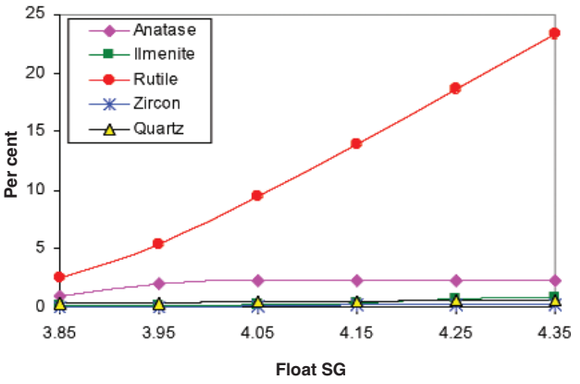

Figure 15—Results of semi-qualitative XRD analysis of accumulative float fractions from Clerici's solution tests (wt%)

Discussion and conclusions

The results show that heavy suspensions of a commercially available non-toxic heavy liquid (LST) and fine heavy particles (tungsten carbide) can effectively separate mineral samples containing high density phases of interest to the sponsoring companies and the wider minerals industry. The heavy suspensions can replace the currently available toxic heavy liquids (diidomethane and Clerici solution), for this purpose.

Density fractionation using heavy suspensions similar to the use of heavy liquid, results in mineral sample concentrates suitable for further detailed analysis (e.g. optical mineralogy or phase/chemical analysis). The experimentation has demonstrated: the separation of rutile from ilmenite, low Ti from high Ti 'ilmenite', zircon from rutile and from ilmenite. Phase fractionation similar to that achieved by a commercial laboratory with Clerici solution has been shown for a –250 + 150 micron rutile-ilmenite-zircon mixture. In addition, the suspension technique has proven capable of zircon separation from ilmenite at higher densities than commercially available Clerici solution and can fractionate zircons into high SG and low SG zircons (clearly related to the levels of metamictization and inclusion content).

While the technique has been shown to be readily applicable to size fractions of particular relevance to the mineral sands industries, viscous effects might prevent the methodology being used at finer sizes or require the further refinement of the technique.

Potential applications of these encouraging results range from the eventual replacement of thallium salts in the laboratory, to industrial gravity separation processes in the production of ultra-pure and specialist products.

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