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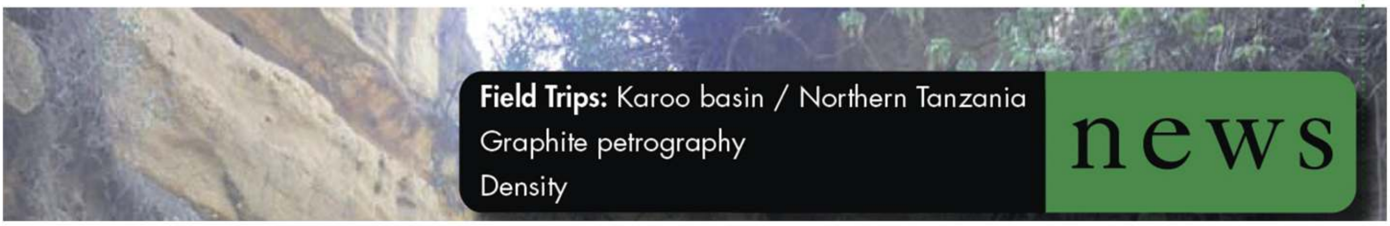

Field Trips: Karoo basin / Northern Tanzania
Graphite petrography
Density

news

density

Industrial Minerals - Why measure Bulk Density?

Introduction

Bulk density is often a relatively neglected parameter during industrial mineral exploration and generally doesn't receive the attention devoted to other measures such as i) sample width in borehole intersections, ii) chemical analysis and iii) product performance testing (Scogings, 2015b).

As noted by Lipton and Horton (2014, p.97) "There are three fundamental inputs to any Mineral Resource estimate: grade, volume and bulk density"; they also state that "The estimation of density commonly receives less attention than is paid to geochemical data and may be based on fewer data points derived from less controlled measurement practices".

Geological resources are generally modelled as volumes in three-dimensional space, after which the estimated volume must be converted to mass using density value/s, thus the measurement of density should be an integral part of the resource estimation process.

The author's intention is to address certain aspects concerning bulk density listed in Section 2.4 of the SAMREC code Table 1. The first part of this review describes bulk density and some of the methods most commonly used for measuring the density of rocks and materials; this is supported by case studies from Minerals Technologies Inc. ("MTI") mines in Australia and South Africa, where the author was previously involved.

The current edition of the SAMREC code for public reporting of Exploration Results, Mineral Resources and Ore Reserves includes Table 1 which is a high-level checklist of assessment and reporting criteria (Table 1). Although not prescriptive, it is important for the Competent Person ("CP") to "report all matters

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SAMREC 2009 Table 1, Section 2.4 Specific gravity and bulk tonnage data

ASSESSMENT CRITERION: T 2.4 Specific gravity and bulk tonnage data
EXPLORATION RESULTS (A)
(i) If target tonnage ranges are reported then the preliminary estimates or basis of assumptions made for bulk density or specific gravity(s) must be stated. (ii) Specific gravity samples must be representative of the material for which a grade rage is reported.
MINERAL RESOURCES (B)
(i) Describe the method of bulk-density / specific-gravity determination with reference to the frequency of measurements, the size, nature and representativeness of the samples.
(ii) The bulk density must have been measured by methods that adequately account for void spaces (vugs, porosity, etc), moisture and differences between rock and alteration zones within the deposit.
(iii) Direct assumptions for bulk density estimates used in the evaluation process of the different materials.
Source: The SAMREC Code 2007, as amended July 2009

that might materially affect a reader's understanding or interpretation of the results or estimates being reported" (SAMREC, page 28). The Code goes further and states that the CP has the responsibility to consider all criteria listed and which additional criteria should apply to the particular project.

The author's intention is to address some of the criteria listed under Section 2.4 'Specific gravity and bulk tonnage data' of SAMREC Table 1 and to provide examples from industrial mineral exploration and mining projects related to the issue of drill core, stockpile and product density. These criteria include the description of "the method of bulk-density / specific-gravity determination with reference to the frequency of measurements, the size, nature and representativeness of the samples" and that "the bulk density must have been measured by methods that adequately account for void spaces (vugs, porosity, etc), moisture and differences between rock and alteration zones within the deposit".

What is Bulk Density?

Bulk density is a measure of mass per unit volume of rock and in South Africa it is generally expressed as metric tonnes per cubic metre. Density is determined by measuring the mass of a sample and dividing this by its volume. Generally the dry mass is obtained by drying the sample and then weighing it, which is the easy part. The challenging bit happens when trying to determine the volume of a rock sample especially when specimens have irregular shapes, are friable, soft and / or porous.

Density may be defined in a number of ways (Table 2) and it is important to ensure that the appropriate density measurement is used for any specific project. Assays for constituents such as Cr_2O_3 (in a chromitite seam), MgO (in magnesite or dolomite) or Graphitic Carbon (in a graphite schist) are normally reported on a dry weight basis and therefore in such cases the 'dry bulk density' (DBD) is applicable. Lipton and Horton (2014) define DBD as the mass per unit volume, including pore spaces but excluding natural water content.

The 'in situ bulk density' (ISBD) includes natural water content and according to Lipton and Horton (2014) should be applied when estimating tonnages of material to be mined. The use of ISBD would apply to a commodity such as bentonite, which may contain 25 to 35% moisture before being mined and core should therefore be sealed immediately after drilling to preserve in situ moisture content.

Specific Gravity (SG) is commonly used to describe density but caution should be exercised, as SG (also known as Relative Density) is often measured using pulverised samples in equipment known as a pycnometer. This method does not take into account porosity or natural water content, which is a limitation of the method for use in geological resource estimation.

Table 2 Description of some commonly used density terms (adapted from Lipton & Horton, 2014)

Term	Units	Definition
Specific Gravity		Relative density: the ratio of the density of the material to the density of water at 4°C
Density	t/m^3	Mass per unit volume
In situ bulk density (ISBD)	t/m^3	Density of the material at natural moisture content
Dry bulk density (DBD)	t/m^3	Density of the material after all free moisture content has been removed

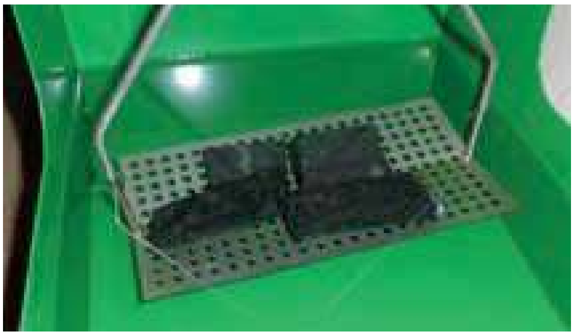
Bentonite core sealed inside plastic to preserve in situ moisture content. Source: MTI

Determining bulk density from small samples

The geologist frequently has only drill core samples to use for density measurement and there are several practical methods available, essentially based around the issue of measuring volume. Each density method has its own potential source of error and it is useful to verify the results of one method against a second if at all possible. It is important to ensure that rock / mineralised samples are representative and that a particular type of rock is not sampled preferentially, e.g. hard material relative to soft material (Lipton and Horton, 2014).

Water displacement method: there are several methods which rely on displacement of water to estimate sample volume and are described in detail by Lipton and Horton (2014, pages 99-101) who list six water displacement methods. One of the most common methods for exploration samples is based on the Archimedes principle in which the sample is first weighed in air, after which it is weighed in water. The density is calculated as the mass of the sample in air, divided by the volume (difference between the sample mass in air and in water). Samples should be competent and not absorb water; if porous they should be waterproofed with substances such as paraffin wax or beeswax which melt at ~60°C, spray lacquer or hairspray, vacuum packed in plastic or wrapped in 'cling wrap' film to help prevent ingress of water.

2 Paraffin wax coated friable chromitite sample being weighed in air. Source: MTI



3 Archimedes (water displacement) equipment. Competent, non-porous chromitite core sample being weighed in water. Source: MTI

Calliper method: this is applicable for drill core samples that can be trimmed at right angles to form a regular cylinder. A pair of callipers is used to measure the core diameter at several points to estimate an average result, while the core length is determined using a tape

4 Paraffin wax blocks. Source: Protea Chemicals, Wadeville

Coating a friable chromitite core sample with molten paraffin wax. Source: MTI

Pyroxenite core sample being measured using a calliper. Source: MTI

Vacuum-packed pyroxenite core. Source: MTI

Pyroxenite core covered with cling wrap, illustrating water ingress. Source: MTI

measure or ruler. The core is weighed and the density determined simply by using the formula of weight / volume. The calliper method has the advantage of simplicity, but it is cautioned that using small diameter core or short core lengths may result in unreliable results (Lipton and Horton, 2014). The calliper method may also be used on half core samples, such as might be encountered when evaluating an older project with previously cut core, or where core has been sampled prior to measuring density. Errors will arise if the core was not aligned correctly when cutting and this should be verified before proceeding with this method. The following parameters should be measured on half core namely i) core length; ii) core segment width and; iii) core segment height (Lipton and Horton, 2014).

Pulp sample method: density of competent rocks that have very low porosity and low natural water content may be measured using a gas pycnometer and rock pulp samples (finely milled rock) but this method is not suitable for porous rocks, as the fabric is destroyed by the milling process. The gas pycnometer method determines volume within the sample chamber from which the gas is excluded. The pycnometer will accurately give volumes for samples weighed into plastic vials which are in turn dropped into the sample chamber. Best precision is obtained from the largest possible volume of sample which is typically around 30 grams. SG data derived from a gas pycnometer may form a useful part of the density database and in the author's experience such SG data can be a valuable QC tool.

Stoichiometric method: there may be an obvious correlation between SG or bulk density and rock chemistry, such as with relatively simple mineral assemblages such as some barite and chromite ores. Assuming that a barite ore consists of discrete barite ($BaSO_4$) and quartz (SiO_2) or that chromitite ore consists essentially of chromite and pyroxene minerals, it should be possible to estimate bulk density based on XRF 'whole rock' analyses. An example is barite ore in which pure barite has a density of ~4.5 g/ml compared with quartz which has a much lower density of ~2.7 g/ml.

Gas pycnometer. Source: Intertek, Perth

Seeing that density is expressed in terms of volume and that XRF whole-rock analyses are expressed on a weight percentage basis, the calculated density must be based on mineral volumes in order to maintain a constant volume. The relationship between whole-rock chemistry and density is non-linear, which is especially obvious when there is a marked difference in density between the different mineral phases (Lipton and Horton, 2014).

Determining bulk density from larger samples

Bulk samples may be obtained if trial mining or production is already in progress at a site. The *in-situ* volume of bulk samples can be estimated by surveying an excavated void (for example an extracted bentonite or chromitite seam) or by surveying a stockpile before and after removal. The sample mass may be determined by directly measuring truckloads across a weighbridge; however sub- samples will have to be taken to determine moisture content as it is impractical to measure the moisture of an entire stockpile or run of mine material. Reconciliation of tonnage mined against the mineral resource or ore reserve is also a good check, not only of the three-dimensional geological model, but also of bulk density.

Operating mines generally measure raw material stockpile volumes for audit and reconciliation purposes, but the question arises of selecting an appropriate bulk density for conversion of volume to mass. Bulk density (BD) values for free-flowing powders and granular materials can vary significantly according to particle size distribution and on how closely the particles are packed. Since powders and granular materials are composed of particles and voids, the volume of a given mass of particles depends on how closely they are packed. In practical terms, the bulk density of a powder tends to increase the more it is subjected to tapping, vibration or other action which causes particles to become better packed, with less void space between larger particles; this is known as the 'tapped bulk density'. Bulk density of free-flowing powders or granular materials can be determined by filling a container of known volume, at which stage the material is weighed and the 'loose bulk density' can be estimated. The container is then tapped and refilled until the material stops settling, at which stage the tapped bulk density can be estimated.

QAQC

QAQC methods commonly applied to other factors in an exploration program such as equipment calibration, duplicates, standards and external laboratory tests should also apply to density measurements.

Case study – chromitite in South Africa

The main aim of this paper is to give practical examples of density measurement and the first example is of drill core from the Batlhako mine at Ruighoek; this MTI operation produces a range of premium-grade chromite sands for foundry, chemical, metallurgical and refractory applications.

In the first example the LG6 chromitite is 'fresh' or un-weathered competent rock consisting predominantly of chromite and pyroxene, hence the Archimedes water

Calibration of a density balance using standard weights. Source: MTI

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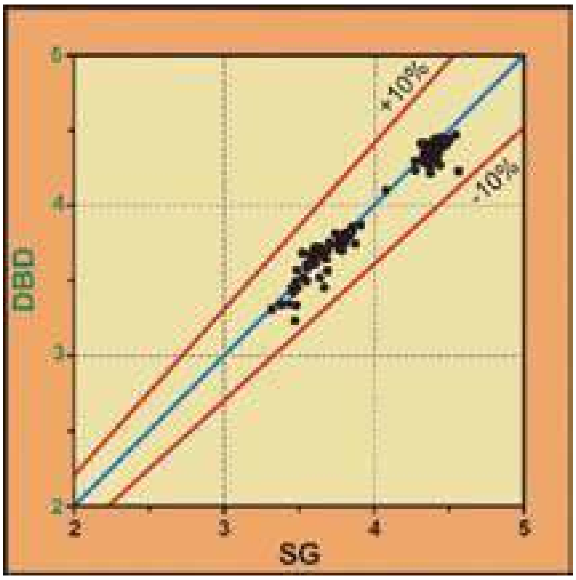
displacement method was deemed suitable. Given that the chromitite seams in this particular example were unweathered, non-porous and competent, a set of milled samples was also analysed by gas pycnometer as a check; this data set demonstrated acceptable correlation between methods.

A further example from Ruighoek concerns pyroxenite drill core from the chromitite hangingwall, which the mine planners wished to evaluate for an open pit situation. In this case the pyroxenite ranged from weathered (friable and porous) to fresh (competent and non-porous) hence there were several options, including water displacement of sealed samples and the calliper method. An un-weathered pyroxenite core sample 'SG6' was chosen as a control and density was estimated using the calliper and various water displacement methods (**Table 3**). The calliper method yielded comparable results to the Archimedes method (uncoated, vacuum packed and paraffin wax). However the 'cling wrap' method proved to be unreliable as it entrained air (reducing the density) and was not waterproof. The author has observed significant differences when using cling wrap on other industrial mineral projects and recommends avoiding this method.



Scatterplot comparing SG (pycnometer) with DBD (Archimedes method) for chromitite and chromiferous pyroxenite. Source: MTI

The density of a second sample of un-weathered pyroxenite core 'SG10' was measured by various methods, after which the core was cut approximately in half and the calliper method used to determine volume. This yielded similar results to the various immersion methods (**Table 4**). Following the initial tests on control sample SG6, a range of pyroxenites and friable chromitites were tested, which illustrated that densities were generally within 1 to 3% of the calliper method. The significantly lower DBD of weathered material (e.g. sample SG2) highlighted the need to test density across a range of weathering domains within a mineral deposit.

It was concluded that for competent, non-porous core samples at the chromite mine the following methods were suitable: i) calliper; ii) water immersion and iii) gas pycnometer, while porous core sample densities are best measured using: i) calliper and ii) wax-coated, spray lacquered or vacuum-packed water displacement methods.

Case study – bentonite in Australia

Measuring the ISBD of sodium bentonite presents a whole set of challenges related to the fact that such material absorbs water and swells; therefore direct immersion in water cannot be used with much confidence.

MTI's sodium bentonite mine is located approximately 350km inland of Brisbane in Queensland, eastern Australia. The bentonite beds were deposited within a high energy fluvial/lacustrine environment of Upper Jurassic to Lower Cretaceous age. Several bentonite beds have been identified on the property; these range up to ~ 4m in thickness and consist of dioctahedral smectite (montmorillonite) with accessory minerals including feldspar, kaolinite, quartz and zeolite. The beds are capped by volcaniclastic rocks identified petrographically as either tuff or ignimbrite in addition to volcanogenic sandstone which is often cross-bedded. Silicified fossil wood is fairly common in sandstones and conglomerates above the bentonite.

In the case of the Australian bentonite example, all exploration drilling was carried out by an open hole method known as Rotary Air Blast (RAB) using a bladed bit, which results in small drill chips unsuitable for water immersion or the calliper method. An alternative drilling method was considered in order to measure ISBD and after discussion with the contractor, the RAB rig was modified to drill core (without water) at several strategic locations. On reclaiming the cores, all samples were sealed in plastic bags to retain in-situ moisture

Table 3 Chromitite and pyroxenite drill core DBD estimated by various methods. Source: MTI

Method	Diameter cm	Length cm	Volume cm^3	Mass in air g	Mass in water g	Sealant mass g	Sealant density g/cm^3	Sealant volume cm^3	Density g/cm^3	Difference vs. Calliper
SG2 oxidised pyroxenite										
Calliper	6.3	11.35	353.95	880.25					**2.49**	
Vacuum pack				887.85	530.75	7.6	0.9	8.26	**2.52**	1.5%
Paraffin Wax				887.35	536.9	7.1	0.9	7.89	**2.57**	3.3%
SG6 competent pyroxenite										
Calliper	4.76	19.6	348.93	1145.85					**3.28**	
Archimedes				1145.85	801.05				**3.32**	1.2%
Cling Wrap				1147.95	786.1	2.1	0.9	2.28	**3.19**	-3.0%
Vacuum pack				1153.5	796.4	7.65	0.9	8.32	**3.29**	0.0%
Paraffin Wax				1154.85	800.2	9	0.9	10	**3.32**	1.2%
11R0007 22.1-22.8m (LG6 chromitite, friable)										
Paraffin wax (1/2 core)				175.8	132.65	1.55	0.9	1.72	**4.21**	
Spray Lacquer (1/4 core)				94.45	71.85	0.4	0.9	0.44	**4.24**	

Table 4 Fresh pyroxenite 'half' core BD estimated using the calliper method. Source: MTI

Sample ID SG10B ('half' core)	Width cm	Height cm	Length cm	Radius cm	Area cm^2	Volume cm^3	Mass in Air g	Density g/cm^3
SG10B fresh pyroxenite ('half' core)								
Calliper	4.72	2.255	18.3	2.36	8.26	151.15	487.55	**3.23**
Archimedes							487.55	3.37
SG10 fresh pyroxenite ('whole' core)								
	Diameter							
Calliper	4.793		18.3	2.40	18.05	330.32	1064.1	**3.22**
Archimedes							1064.1	3.35

before estimating density. The core samples were then trimmed with a hacksaw to yield regular cylindrical shapes from which volumes could be estimated using the calliper method, and moisture content derived from the 'shavings'. Density values of between 1.72 and 1.84 t/m3 were obtained (Table 5) and it was elected to use 1.8 t/m3 for estimation of *in-situ* 'wet' bentonite resources.

Once a mine is in operation, it is advisable to verify densities that were estimated during the exploration phase of the project. This can be achieved by surveying the volume of an excavated void, for example an extracted bentonite seam in an opencast

RAB drill chips at the Australian bentonite mine. Source: MTI

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10 Bentonite core trimmed for calliper method. The core 'shavings' were used for moisture analysis. Source: MTI

pit and using this in conjunction with truckloads of mined material measured on a weighbridge (Table 6). This procedure was adopted at the Australian mine and verified that an ISBD range of 1.74 to 1.8 t/m3 is probably applicable to this type of bentonite (~27% moisture; ~ 80% montmorillonite). It is to be expected that ISBD would vary across such a deposit according to mineralogical composition, degree of weathering, moisture content and overburden thickness. A further benefit of reconciling actual volume and tonnes mined against the estimated mineral resource volume and tonnes is to verify the geology model. In this particular case the surveyed volume was within 3% of the modelled volume, indicating that the exploration and modelling methods were applicable for this style of bedded mineralisation.

Table 5 Bentonite ISBD estimated using the calliper method. Source: MTI

Bentonite	Length cm	Diameter cm	Volume cm^3	Mass g	Moisture %	ISBD tonnes $/m^3$
5D	4.6	6.4	148.0	254.6	27.3	**1.72**
5D	2.3	6.4	74.0	135.9	27.0	**1.84**

Another example from the Australian mine addresses the estimation of bulk density of sun-dried (granular) bentonite stockpiles. As with surveying the volume of bentonite mined from a pit, an option for stockpiles is to measure the stockpile before and after shipment and estimate the volume removed. An alternative is to extract some material from the stockpile and fill a container of known volume, which can then be weighed. This latter procedure was adopted at the Australian bentonite mine and it was estimated from filling a box of one cubic metre volume that; i) loose (untapped) density was ~1.3 t/m3 and ; ii) that tapped density is ~1.4 t/m3 (**Table 7**).

Table 6 Bentonite ISBD estimated from a surveyed open pit, Australia. Source: MTI

Description	Bentonite	Tonnes hauled (over weighbridge)	Volume m^3 (surveyed)	Volume m^3 (geology model)	Difference in volume	ISBD (calculated t/m3)
NP Block 3	5D	10,017	5,773	5,947	3%	**1.74**

Case study - Barite

Given the recent trend towards the use of lower SG barite for oil drilling applications, this study evaluated the stoichiometric method of estimating density using a series of barite-silicate blends. The American Petroleum Institute standard for oil drilling barite was SG 4.2 until 2010, a new lower SG 4.1 product was accepted as an alternative standard. Over the past few years some experts in the oil industry expressed reservations that, as lower SG is related to dilution of barite by abrasive contaminants such as 'silica' (quartz or chert); this

'Dried and crushed' bentonite about to be tipped into cubic metre box. Source: MTI (above and below)

Table 7 Bentonite stockpile BD measured using a cubic metre box. Source: MTI

Bentonite	Location	Untapped Mass (tonnes)	Tapped Mass (tonnes)	Volume (m^3)	Moisture (%)	Density Untapped t/m^3	Density Tapped t/m^3
5B OP	Dry Stockpile	1.253	1.41	1	10.4	**1.25**	**1.41**
5D OPW	Dry Stockpile	1.306	1.43	1	11.9	**1.31**	**1.43**

would result in increased mill wear, increased tonnage required to be milled and more abrasive drilling mud. The consensus was that going to an even lower 4.0 SG standard would release more barite into the market, but could cause problems and increased costs for drilling fluids and waste management (Scogings, 2015a).

The author has calculated SG for a theoretical series of barite-quartz compositions between SG 2.7 and SG 4.5 in an attempt to quantify the effect of dilution by 'silica contaminants'. In addition, a series of barite-quartz dilutions (by mass) were prepared and measured by gas pycnometer at Intertek in Perth, Australia. The pycnometer results appear to verify i) the non-linear relationship between whole-rock chemistry and density and; ii) that a barite product with density of 4.1 could have as much as 23% silicate mineral by volume, rising to ~30% when SG is decreased to 4.0. This latter value for mineral impurities at SG 4.0 is higher than when assuming a straight-line relationship between chemistry and SG (**Table 8**).

Conclusions

- Mineral resource estimations rely on three main inputs: i) grade, ii) volume and iii) bulk density, of which the latter is often relatively neglected during mineral exploration

- The SAMREC 2009 code requires that the methods and assumptions of estimating bulk density be described when reporting Mineral Resources and Reserves

- Poor quality bulk density measurements result in unreliable tonnage estimates and impact negatively on mine scheduling and reconciliation of mineral production against reserves

- Determination of sample mass is the 'easy part' of estimating density. The difficult step generally lies in trying to determine the volume of a sample

- There are several methods for estimating the volume of rocks and materials, each of which has practical limitations and it is suggested that more than one method be used, as an internal check

Table 8 Barite product:
SG related to dilution by SiO_2 impurities

$BaSO_4$ (% by mass)	SiO_2 (% by mass)	SG (calculated) (g/mL by mass)	Barite (calculated) (% by volume)	Silicate (calculated) (% by volume)	SG (calculated) (g/mL by volume)	SG (pycnometer) (g/mL lab. blend)
100	0	4.50	100	0	4.50	4.50
95	5	4.41	92	8	4.35	4.38
90	10	4.32	84	16	4.22	4.22
85	15	4.23	77	23	4.09	4.04
80	20	4.14	71	29	3.97	4.01
75	25	4.05	64	36	3.86	3.78
50	50	3.60	38	63	3.38	3.46
25	75	3.15	17	83	3.00	3.02
0	100	2.70	0	100	2.70	2.71

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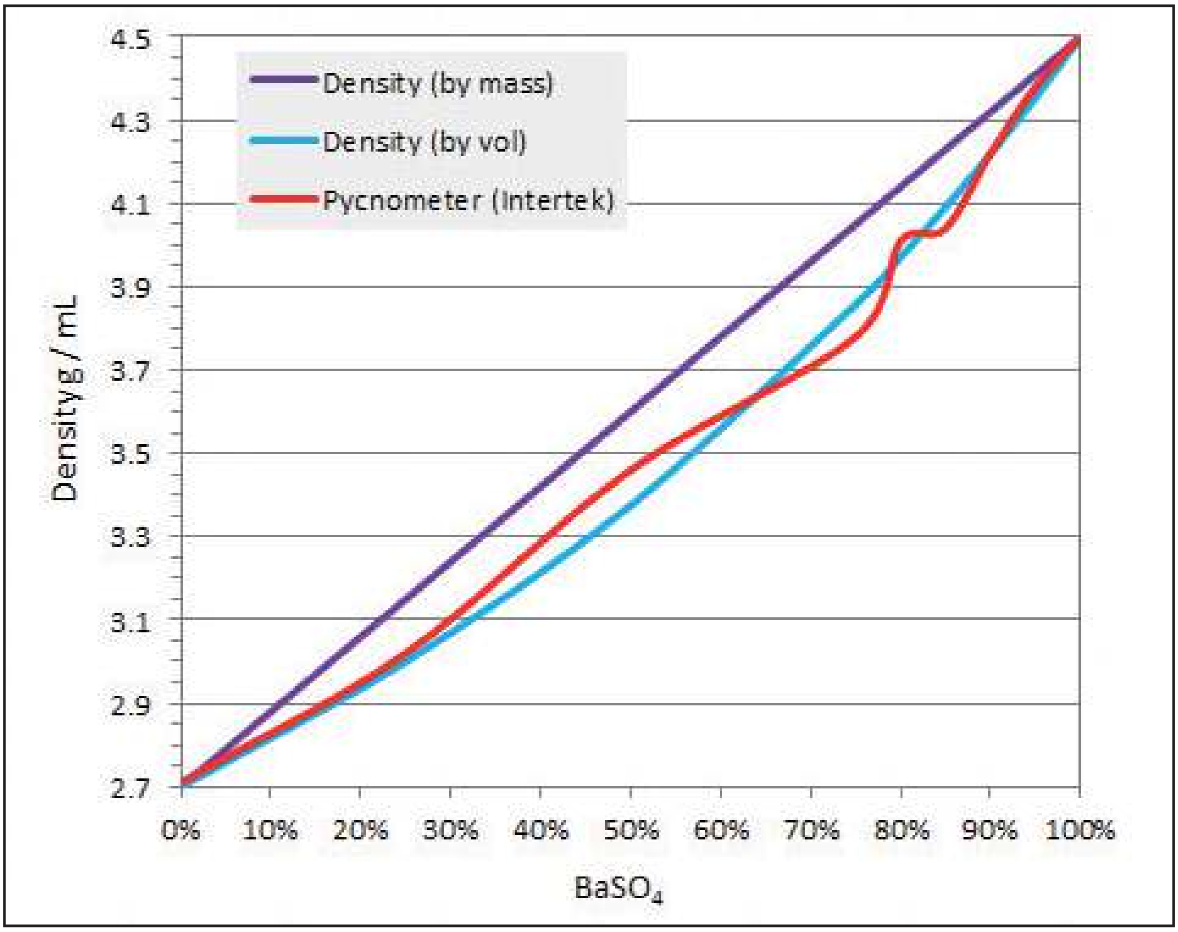

Stoichiometric density estimates for barite-quartz blends. Source: Industrial Minerals Research, Intertek Perth

- The use of ‘cling wrap’ film to seal samples should be avoided, as entrapped air can lead to significantly low density results compared with other methods

- The method/s chosen should take into account physical and chemical variations across the deposit such as weathering, porosity, mineralogy and moisture content

- QAQC methods commonly applied to other factors in an exploration program should also apply to density measurements.

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