Precise Pb–Pb baddeleyite ages of 1765 Ma for a Singhbhum 'newer dolerite' dyke swarm

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The Singhbhum craton in eastern India hosts a number of mafic dyke swarms popularly called 'newer dolerites'. Previous attempts to obtain emplacement ages of these rocks were limited to a few poor-precision K-Ar whole-rock and Rb-Sr isochron ages. Here, two prominent dykes from the WNW-ESE trending swarm in the south central region of the craton were dated using Pb-Pb baddeleyite thermal extraction-thermal ionization mass spectrometer method. These dykes yielded identical baddeleyite Pb-Pb ages of 1766.2 ± 1.1 Ma (SKJ-10) and 1764.5 ± 0.9 Ma (SKJ-15) respectively, which are interpreted as the time of emplacement of the WNW-ESE trending 'newer dolerite' dyke swarm. The predominantly parallel dyke trend in this swarm for over 100 km along strike indicates these dyke fractures were formed due to horizontal compressive stresses in a region that may have been associated with a palaeo compressional system.

Coeval ~1770 Ma magmatism in the Singhbhum craton and in China, Australia, Brazil and Uruguay confirms this event was globally widely dispersed. The timing of this event also coincides with orogenic activity in majority of continents that may have formed during the assembly of supercontinent Columbia.

Keywords: Baddeleyite ages, coeval magmatism, cratons, dyke swarms.

EARTH's history is punctuated by numerous periods when large volumes of mafic magma were emplaced. Magmas genetically unrelated to seafloor spreading and subduction are termed large igneous provinces (LIPs) and consist of continental flood basalts^{1,2}. LIPs of Palaeozoic and Proterozoic age are usually deeply eroded, and occur as giant dyke swarms, sill provinces and layered intrusions. Many LIPs have been linked to regional-scale uplift, continental rifting and break-up, and climatic crises³. Their ability to retain high-quality U–Pb geochronological, palaeomagnetic and geochemical data makes them ideal subjects for reconstructing past continental configurations and therefore for addressing the concept of repeated supercontinent formation and disaggregation⁴.

Spectacular dyke swarms known as 'newer dolerites' occur within the Singhbhum granite complex in eastern

India (Figure 1). Petrologically these vary from doleritic, leucogranophyric to ultramafic/noritic in composition. Doleritic dykes are however the more dominant (99%) type and outnumber the other two. The newer dolerites were emplaced in four distinct strike patterns, NNE-SSW, N-S, NNW-SSE and WNW-ESE. Many age determinations have been attempted on these dykes. The K-Ar whole-rock ages vary considerably within and between swarms and range from 2144 to 950 Ma (refs 5-7). While a distinctly older Rb-Sr whole-rock isochron age of 2613 ± 177 Ma (ref. 8) was reported for a NNE-SSW trending ultramafic dyke. Although these age determinations have shown that most of the newer dolerite swarms are Proterozoic and at least one of these could be late Archaean, it is not clear whether the large spread in the measured ages from Mesoproterozoic to Neoarchaean is real or due to inherent limitations of the dating methods when applied to Proterozoic rocks.

Precise U–Pb age determination of mafic magmatism (to $\sim 0.1\%$ precision) has been possible mainly by the analysis of baddeleyite (ZrO₂) found as a common accessory mineral in these rocks. Baddeleyite generally



Figure 1. Simplified geology map of a part of the Singhbhum craton (adapted from the Geological Survey of India map³⁴ and dyke distribution after *Google Earth* satellite images) showing 'newer dolerite' dyke swarms. Stars indicate site locations of samples used for Pb–Pb age determinations in the present study. WNW striking dykes are shown in black and dykes of other orientations are shown in green.

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contains sufficiently high concentrations of uranium (500 to several 1000 ppm)⁹ with concordant or nearly concordant (less than 1% discordant typically) U–Pb data even without any of the pre-treatments typically required for zircon preparation, as it appears to be much less susceptible to Pb loss than zircon^{9–13}, and importantly, it rarely occurs as xenocrysts in these rocks. Due to such exceptional qualities, this mineral is considered to be an ideal geochronometer for U–Pb or Pb–Pb dating of mafic and ultramafic rocks. The present communication presents $^{207}Pb/^{206}Pb$ baddeleyite age determinations on two promi-

nent dykes from the WNW–ESE trending 'newer dolerite' dyke swarm (Figure 1) using the thermal extraction–thermal ionization mass spectrometer (TE–TIMS) approach, and also reported in Davis¹⁴ and Kumar *et al.*¹⁵.

For separating baddeleyite, samples (~200 g) were crushed to <125 µm grain size and concentrated using a Wilfley table, followed by handpicking under a binocular microscope. Best quality (mineral inclusions, fractures and alteration-free) baddeleyite crystals were cleaned in 4N HNO₃ and were rinsed several times in MQ (Milli-Q) H_2O and dried. Zone-refined rhenium filaments (0.030 inches wide by 0.001 inches thick) used for loading the baddelevite were welded on the posts and bent using a jig to form a transverse 0.5 µm broad and 0.5 µm deep U-shaped valley in the centre. These were outgassed and cleaned at alternating high (4.0 A) and low (2.0 A)temperature steps with the complete sequence lasting for 90 min, and left for at least a week before loading. After washing with HNO₃ and ultraclean water (MQ), the grains were loaded onto filaments for annealing at 1250°C in a vacuum chamber at $\sim 1 \times 10^{-7}$ mbar for 30 min to expel disturbed Pb from altered domains. Grains were subsequently embedded in silica glass on the same Re filament by fusing the sample with silica gel and phosphoric acid mixture at 1200°C for ~10 min in a vacuum chamber. Sample loading was performed under a stereomicroscope in a clean air station fitted with a HEPA filter. Analyses were performed on a TRITON^{PLUS} TIMS at the CSIR-National Geophysical Research Institute, Hyderabad. The three high-mass Faraday detectors (H1 to H3) connected to 10¹¹ ohm resistors were used to measure ²⁰⁶Pb, ²⁰⁷Pb and ²⁰⁸Pb, in static multi collector mode with virtual amplifier rotation. The ²⁰⁴Pb signal was very small and therefore was collected simultaneously by a secondary electron multiplier-ion counting system on the axial channel. Isotopic ratios were corrected for instrumental mass fractionation of 0.18%/atomic mass unit. This value is marginally higher than the average of 0.1%generally observed with Pb emission for dissolved silica gel loads16

The ²⁰⁷Pb/²⁰⁶Pb ages for each block were calculated from the determined ²⁰⁷Pb/²⁰⁶Pb and ²⁰⁴Pb/²⁰⁶Pb ratios with corrections for common Pb, according to the model proposed by Stacey and Kramers¹⁷. Ages are determined from weighted means of fraction averages using accepted

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decay constants¹⁸. Details regarding data acquisition and data reduction are given in Kumar *et al.*¹⁵.

Five baddeleyite fractions from Phalaborwa carbonatite standard analysed during this work yielded a 207 Pb/ 206 Pb weighted mean age of 2060.1 ± 0.7 Ma, with an MSWD (mean square of weighted deviates) of 0.57 (Table 1 and Figure 2). This age is indistinguishable (within error) from the reported 207 Pb/ 206 Pb age of 2059.7 ± 0.35 Ma (MSWD = 7.7, *n* = 59) by Heaman¹⁹ and 2060.6 ± 0.5 Ma by Reischmann²⁰, obtained by the conventional U–Pb isotope dilution TIMS method.

Pb isotopic results for five baddeleyite fractions each separated from SKJ-10 and SKJ-15 are given in Table 1 and shown in Figure 3. Fraction weighted mean ²⁰⁷Pb/ ²⁰⁶Pb ages for the two samples SKJ-10 and SKJ-15 are 1766.2 ± 1.1 Ma (MSWD = 1.6) and 1764.6 ± 0.9 Ma (MSWD = 2.1) respectively. If the assigned errors are the only cause of scatter, MSWD will tend to be close to 1 (ref. 21). In these datasets the MSWD is marginally higher at 1.6 and 2.1 respectively, and cannot be accounted by analytical errors alone. The higher MSDW for TE-TIMS data is attributed to fractionation error, which was estimated to be 0.05% for baddeleyite analysis¹⁵. Adding a 0.05% (0.88 Ma) error to the data resulted in 1766.0 ± 1.0 Ma age (MSWD = 0.62) for SKJ-10 and 1764.6 ± 0.9 Ma age (MSWD = 0.64) for SKJ-15, reducing the MDSW to <1 but resulting in the same errors for the weighted mean ages, suggesting that fractionation error should be included in all TE-TIMS data. Therefore, the grand mean ages of 1766.0 \pm 1.0 and 1764.6 \pm 0.9 Ma are interpreted as the best estimate for baddeleyite crystallization in these dykes and hence the emplacement age of the WNW-ENE trending 'newer dolerite' dyke swarm.

Reported K–Ar ages on this dyke swarm are considerably lower and vary between 1241 and 1264 Ma (ref. 7).



Figure 2. TE-TIMS weighted mean ²⁰⁷Pb/²⁰⁶Pb age of five baddeleyite fractions from the Phalaborwa carbonatite standard. Errors bars represent 95% confidence limits of measurements.

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Sample no.	Sample weight	No. of blocks	²⁰⁶ Pb/ ²⁰⁴ Pb (m)	Absolute error	²⁰⁷ Pb/ ²⁰⁶ Pb (m)	²⁰⁷ Pb/ ²⁰⁶ Pb (c)	Pb–Pb age (Ma)
SK I- 10							
SKI 10-1	49	24	64053	476	0.108139 ± 226	0.107973 ± 040	1765 7 + 2 4
SKI 10-2	5.1	26	22591	311	0.108593 ± 160	0.107996 ± 0.032	1766.8 ± 1.8
SKI 10-3	5.0	15	29623	293	0.108749 ± 161	0.107996 ± 0.002	1766.8 ± 2.6
SKI 10-4	5.0	37	42363	649	0.108325 ± 0.004	0.107960 ± 0.050	1765.2 ± 2.6
SKI 10-5	5.1	28	41146	696	0.108228 ± 0.095	0.107907 ± 0.050 0.107907 ± 0.052	1763.2 ± 2.0 1764.7 ± 2.8
Grand weighte	d mean after adding f	fractionation	uncertainty of 0.04	55%	0.100220 ± 095	0.107907 ± 032	1701.7 ± 2.0
Age: 1766.0 ±	1.0 Ma (MSWD = 0.)	62)	i uncertainty of 0.02	5570			
SKJ-15							
SKJ 15-1	4.8	20	14807	191	0.108467 ± 126	0.107944 ± 031	1765.5 ± 1.9
SKJ 15-2	4.7	30	15417	144	$0.108835 \pm 0.0000000000000000000000000000000000$	0.107976 ± 029	1765.4 ± 2.2
SKJ 15-3	4.6	30	19812	282	0.108457 ± 061	0.107881 ± 028	1763.6 ± 1.9
SKJ 15-4	4.5	30	21270	302	0.108504 ± 047	0.107894 ± 023	1764.4 ± 1.7
SKJ 15-5	4.6	30	18451	253	0.108559 ± 055	0.107907 ± 0.039	1764.6 ± 2.2
Grand weighte	d mean after adding f	fractionatior	uncertainty of 0.05	55%			
Age: 1764.6 ±	0.9 Ma (MSWD = 0.	64)	, i i i i i i i i i i i i i i i i i i i				
Phalaborwa ca	rbonatite (BD-1)						
BD 1-8	12.4	17	26661	1654	0.127734 ± 032	0.127198 ± 041	2059.6 ± 1.5
BD 1-9	11.6	14	12855	543	0.128296 ± 068	0.127195 ± 044	2059.6 ± 1.5
BD 1-10	12.0	16	10315	829	0.128566 ± 0.91	0.127230 ± 028	2060.0 ± 1.5
BD 1-11	5.8	25	23056	392	0.127862 ± 043	0.127252 ± 042	2060.4 ± 1.5
BD 1-12	5.8	22	26882	489	0.127799 ± 043	0.127292 ± 032	2060.9 ± 1.5
Grand weighte	d mean after adding f	fractionatior	uncertainty of 0.05	55%			
Age: 2060.1 ±	0.7 Ma (MSWD = 0.)	57)	-				

Table 1. TE-TIMS Pb isotopic data on baddeleyite fractions from the newer dolerite dykes and Phalaborwa carbonatite baddeleyite standard

Sample weight is in micrograms. $^{206}Pb/^{204}Pb$ (m) and $^{207}Pb/^{206}Pb$ (m) are measured values (fraction means with standard error) and $^{207}Pb/^{206}Pb$ (c) is the corrected value (weighted means and errors). Uncertainties are $2\sigma_m$ and refer to the least significant digits.



Figure 3. TE-TIMS weighted mean 207 Pb/ 206 Pb age of five baddeleyite fractions from Singhbhum 'newer dolerite' dyke samples, (a) SJK-10 and (b) SJK-15. Errors bars represent 95% confidence limits of measurements.

Close observation at dyke intersections indicates that the dated WNW–ENE trending 'newer dolerite' dyke swarm distinctly cuts dykes of other orientations at several locations, suggesting this could be one of the youngest swarms in the region. If the older Rb–Sr whole-rock isochron age of 2613 ± 177 Ma (ref. 8) reported for a NNE–SSW trending ultramafic dyke is accepted as its emplacement age, within its large error limits (subject to refinement by U–Pb or Pb–Pb baddeleyite/zircon method), these two swarms which constitute a large proportion of the 'newer dolerite' dykes in the Singhbhum craton were emplaced during the Palaeoproterozoic and Neoarchaean respectively. K–Ar whole-rock ages on the other hand, are considerably younger and vary from 2144 to 950 Ma (refs 5–7). If the Pb–Pb baddeleyite and Rb–Sr (within their large errors) ages are accepted as their actual emplacement ages (considering that the present authors unpublished Pb–Pb baddeleyite age for the older dyke swarm is also similar within errors to the Rb–Sr age), the 'newer dolerites' were emplaced ~700 Ma earlier. This could have important geological implications, as many correlations were based on newer dolerite ages. This may even necessitate a revision in the Proterozoic stratigraphy of the Singhbhum craton. Additional baddeleyite geo-chronology on other dyke swarms from the region has been initiated to substantiate this inference.

Though the 1765 Ma age reported here is the precise Pb–Pb baddeleyite age for a Singhbhum dyke swarm, several dyke swarms have been precisely dated using the U-Pb baddeleyite/zircon method from the Dharwar, Bastar and Bandelkhand cratons. A first-order comparison of dyke events between these cratons shows the oldest known and perhaps the most dominant swarm in the Dharwar craton is the 2367 Ma Dharwar giant dyke swarm, with an aerial extent of nearly the entire eastern Dharwar craton^{4,22}. Other dated dyke swarms from the eastern Dharwar craton include a N-S-oriented swarm at 2220.5 \pm 4.9 Ma, a NW–SE striking swarm at 2209.3 \pm 3.8 Ma and a radial WNW-ESE to NW-SE swarm at 2180.8 ± 0.9 to 2176.5 ± 3.7 Ma (refs 23–25). In addition, a large igneous province emplaced between 1891 and 1883 Ma consisting of mafic dykes in the Bastar craton and mafic-ultramafic sills in the Cuddapah basin was recognized by French et al.²⁶. Two dyke swarms emplaced at 1979 ± 8 and 1113 ± 7 Ma have been reported from the Bandelkhand craton²⁷. A younger $1466.4 \pm$ 2.6 Ma dyke swarm has been reported from the Lakhana region in the Bastar craton²⁸. Except for the 1885 event in the Bastar and Dharwar cratons, there are no matching events between them. This suggests that the number of precisely dated events from either of these Indian cratons is inadequate for a meaningful comparison between them or to speculate on their ancestry within other proto cratons/continents.

Though dyke swarms of 1765 Ma age have not been reported from other Indian cratons, coeval mafic magmatism is known from several other regions, including China, North China craton $(1769 \pm 2.5 \text{ Ma}, \text{ U-Pb}, \text{ zircon})^{29}$, Kimberley basin in Australia $(1790 \pm 4 \text{ Ma}, \text{ U-Pb}, \text{ zircon})^{30}$, Avanavero, Roraima, Brazil, $(1794 \pm 4 \text{ Ma}, \text{ U-Pb}, \text{ Baddeleyite})^{31}$, Uruguay (Rio de Plata Craton, $1790 \pm 5 \text{ Ma}, \text{ U-Pb}$, Baddeleyite)³². The timing of this event coincides with orogenic activity in several continents which could have formed during the assembly of a supercontinent Columbia³³. The WNW–ESE dyke swarm studied here could also be an expression of the same orogenic activity.

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