A new precise date for the Tolmie Igneous Complex in northeastern Victoria

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Among the Devonian igneous complexes of central and northeastern Victoria, the S-type Tolmie Igneous Complex (TIC) occupies a unique spatial position, astride the Governor Fault and the Mount Wellington Greenstone Belt, a probable terrane boundary, and also bridges the boundary between the Strathbogie and Tabberabbera granite provinces. This stitching position makes it important to determine the nature of the crustal source involved in its genesis. Here we present a new U/Pb zircon age for a sample of the Toombullup Ignimbrite, which forms a large part of the TIC. This age (377 ± 2 Ma) allows us to comment far more reliably on the initial Sr and Nd isotope ratios of the TIC magmas. The corrected data (initial 87Sr/86Sr D0.70804 to 0.71085, εNd D0.08 to 0.44) confirm a heterogeneous sediment-dominated protolith, but unlike those of other S-type granitic and felsic volcanic rocks of the region, one composed of materials that had not long previously been extracted from the mantle. This finding has importance in the context of future models for the geological and tectonic development of this part of southeastern Australia.

KEY WORDS: U/Pb dating, zircon, Late Devonian, Tolmie Igneous Complex, Toombullup Ignimbrite, rhyolite, Victoria, Australia.

INTRODUCTION

The Tolmie Igneous Complex (TIC) proper consists of several S-type, rhyolitic ignimbrite sheets within the Wabonga Cauldron (Gaul 1995), together with a number of small hypabyssal stocks of porphyritic microgranodiorite. The TIC is intruded at its northwestern end by what was formerly known as the Barjarg granite but is now recognised as a part of the Lima pluton of the Strathbogie batholith (Phillips & Clemens 2013). All these igneous rocks are Late Devonian in age and form part of the Central Victorian Magmatic Province (VandenBerg et al. 2000). This felsic-dominated magmatism occurred in a post-orogenic extensional regime. The TIC lies 220–250 km northeast of the city of Melbourne, forming the northern end of the Howitt Province, a NW–SE-trending belt of Upper Devonian, non-marine, sedimentary and volcanic rocks most probably deposited in a transtensional basin (VandenBerg et al. 2000). The position of the TIC within the context of the geology of Victoria is illustrated in Figures 1 and 2.

Clemens et al. (2011) found that the ignimbrites contain multiple generations of phenocryst assemblages. The Toombullup Ignimbrite contains early phenocrysts that crystallised at mid-crustal pressures of about 400 MPa and at temperatures >850°C (with indications of temperatures nearer 900°C). The Toombullup Ignimbrite also contains a later microphenocryst suite (including quartz, ferroan orthopyroxene and fayalitic olivine) that formed at subvolcanic pressures of < 150 MPa. The underlying Ryans Creek Ignimbrite (RCI) contains garnet phenocrysts with rutile inclusions, indicating earliest crystallisation at deep crustal conditions (around 35 km depth). Clemens et al. (2011) interpreted the geochemistry and isotope systematics of the TIC rocks to indicate derivation through partial melting of metamorphosed arc greywackes containing variable proportions of clay. Clemens et al. pointed out that the deep origin of some of the TIC magmas means that supracrustal rocks underlie this part of Victoria at depths of around 35 km. Thus, the Paleozoic metasedimentary rocks were not deposited on ocean-floor material, but rather must overlie probable Proterozoic crust of the Selwyn Block (Cayley 2011).

EXISTING CONSTRAINTS ON THE AGE OF THE TIC

Since it was not deformed in the Tabberabberan Orogeny and unconformably overlies the folded and eroded Paleozoic rocks, the TIC must have been emplaced after the end of the orogeny; which spanned the period from 450 to 395 Ma in this region. McDougall et al. (1966) published an Rb–Sr age of 368 Ma for the RCI, which forms the lowermost ignimbrite unit in the TIC proper. This age used an old and incorrect decay constant and was also derived using an assumed initial Sr isotope ratio of 0.704, far too low for an S-type, crustally derived magma.

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Using the McDougall et al. (1966) data with a more realistic initial ratio of 0.710 ± 0.002, and with the correct decay constant, the calculated age is 377 ± 27 Ma, not a particularly precise date. Paleontological evidence from Long & Werdelin (1986) shows that the Lewis Farm Conglomerate, which underlies the TIC, may be as old as Givetian (392 to 385 Ma), and the upper volcanic unit of the TIC, the Toombullup Ignimbrite is intruded by the Lima pluton of the Strathbogie batholith (Phillips & Clemens 2013), dated at 366 ± 7 Ma by Richards & Singleton (1981), using the Rb–Sr method. Bierlein et al. (2001) and Kemp et al. (2008) published SHRIMP U–Pb zircon ages of 374 ± 2 and 366 ± 3 Ma, respectively, for samples of Strathbogie granite, from the Mount Wombat pluton. Clemens et al. (2011) used the existing constraints (although omitting the Bierlein et al. date for the Strathbogie batholith) to suggest that the main phase of the TIC was emplaced at 375 ± 17 Ma. Again, this is not a particularly precise date, and its accuracy could also be questioned.

THE PROBLEM

Clemens et al. (2011) obtained Sm–Nd and Rb–Sr isotope data for rocks of the TIC and used these to calculate initial ɛNd and initial 87Sr/86Sr values for the magmas, with the aim of constraining the character of their deep-crustal protoliths and helping to characterise the mechanisms by which the geochemical variations came about. Unfortunately, these aims were hampered by the imprecision in the existing data for the TIC (± 17 Ma), leading to large errors in the calculated initial isotope ratios, particularly for the Rb–Sr system. Clemens et al. (2011, p. 1330) wrote 'The TIC rocks have a range of initial 87Sr/86Sr values (0.70734–0.71067). However, the age uncertainty results in large errors on these values and little can be concluded from these data beyond the confirmation that the magmas had an evolved crustal source.' Also, among the Devonian igneous complexes of this region, the TIC sits in a unique position, astride the Governor Fault and the Mount Wellington Greenstone Belt of tholeiitic to boninitic rocks. This belt is probably a terrane boundary and the TIC also straddles the more complex geochemical boundary between the Strathbogie and Tabberabbera granite provinces (Rossiter 2003), inferred to mark a junction between two contrasting sets of deep crustal protoliths for the Devonian felsic magmatic rocks of the region (see also, Rossiter & Gray 2008). For these reasons we considered it important to obtain a better-constrained date for the TIC.

APPROACH

To obtain a more precise and accurate date for the TIC, we used laser-ablation ICP-MS analyses of zircon crystals separated from a sample of Toombullup Ignimbrite (Toom 1) collected from 36°46.1891′S, 146°21.6087′E, at an elevation of 738 m ASL, at the side of the C521 Mansfield-Whitfield Road, 5.5 km SE of the town of Myrrhee and 4.5 km W of Whitfield, in the Tolmie Highlands of Victoria. This sample comes from the upper part of the Toombullup Ignimbrite, where it is most mafic. Table 1 shows the composition of this sample, determined by XRF analysis at the University of Salzburg.

Zircon separation

The rock was crushed in a jaw crusher to a grainsize of <2 mm then passed through a 400 μm sieve. The fraction passing the sieve was further processed on a wet shaking table to obtain a heavy-mineral concentrate. A representative split of this concentrate was mounted on a glass slide and embedded in Araldite for transmitted light microscopy. More zircons were handpicked under the
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microscope, placed onto a planar glass surface and fixed in a disk of epoxy resin. After grinding the zircons down to approximately half their width, cathodoluminescence images were taken using a Zeiss ULTRAPLUS scanning electron microscope at the University of Salzburg. Based on these images, we identified zircon domains with uniform CL texture, free of cracks and alteration. These were selected and labelled for LA-ICP-MS analysis.

Figure 2 Simplified geological map of the green-shaded area in Figure 1, based on VandenBerg et al. (2000, figure 2.113), showing the main geological units associated with the Tolmie Igneous Complex (TIC). Note that the TIC lies on the northwestern end of the chain of volcanic and red-bed basins that straddle the Governor Fault, which is inferred to be the surface expression of a terrane boundary, separating the Melbourne and Tabberabbera zones. For a more detailed map of the TIC the reader is referred to Clemens et al. (2011, figure 1 and electronic supplementary material ESM 1).
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Table 1 Composition* of sample Toom 1 from the Toombullup Ignimbrite.

| SiO₂    | 71.81 | Ba | 1771 | Ni | 11 | Yb | 7  |
|---------|-------|----|------|----|----|----|----|
| TiO₂    | 0.44  | Ce | 200  | Pb | 38 | Zn | 94 |
| Al₂O₃   | 14.06 | Co | 5    | Rb | 128| Zr | 320|
| FeO⁷    | 3.04  | Cr | 17   | Sc | 9  |    |    |
| MnO     | 0.06  | Dy | 11   | Sr | 120|    |    |
| MgO     | 0.99  | Ga | 20   | Th | 30 |    |    |
| CaO     | 2.05  | Gd | 16   | U  | 4  |    |    |
| Na₂O    | 2.88  | La | 89   | V  | 43 |    |    |
| K₂O     | 4.47  | Nb | 16   | W  | 16 |    |    |
| P₂O₅    | 0.14  | Nd | 84   | Y  | 80 |    |    |

*Major oxides normalised to 100 wt% anhydrous, with all Fe expressed as FeO⁷ and trace elements in ppm by weight; the values for Gd, Dy and Yb are approximate but of the correct order.

LA-ICP-MS analytical details

All U–Pb age data obtained at the Central Analytical Facility of Stellenbosch University, were acquired by laser ablation–single collector–magnetic sector-field–inductively coupled plasma–mass spectrometry (LA-SF-ICP-MS), employing a Thermo Finnigan Element2 mass spectrometer coupled with a Resonetics Resolution S155 excimer laser ablation system. All age data presented here were obtained from single-spot analyses with a spot diameter of 30 μm and a crater depth of approximately 15 to 20 μm. The methods employed for analysis and data processing closely followed the procedures described by Frei & Gerdes (2009) except that, in this study, an excimer laser source was used for ablation. GJ-1 was used as calibration standard. To monitor the 206Hg interference on 204Pb, mass 202Hg was measured and used to correct significant common-Pb contributions using the model by Stacey & Kramers (1975). The 206Hg/204Hg ratio used is 4.36. Common Pb corrections were applied only in cases where 204Pb counts statistically exceeded average background levels. In these corrections, we assumed a 207Pb/206Pb ratio of 0.83, equivalent to the present-day ratio of terrestrial Pb isotopes in the Stacey & Kramers (1975) model. For quality control, the Plesovic (Aftalion et al. 1989; Sláma et al. 2008) and M127 (Nasdala et al. 2008; Mattinson 2010) zircon reference materials were analysed with the unknowns, and the results were consistently in excellent agreement with the published ID-TIMS ages. Full analytical details and the results for all quality-control materials are reported in Supplementary Papers (Table A1, A2). The calculation of concordia ages and plotting of concordia diagrams were performed using Isoplot/Ex 3.0 (Ludwig 2003).

RESULTS

Zircon typology

In transmitted light, two zircon groups can be distinguished. Approximately half of the crystals have morphologies characterised by dominant {110} and {211} faces. These major faces typically occur in combination with minor {100} and {101} faces. Crystals with this morphology (group I in Figure 3) are generally considered typical of zircons in peraluminous, crust-derived granitic rocks (Pupin 1980), and particularly in S-type granites (Schermaier et al. 1992).

The second group of zircons (group II in Figure 3) has large {100} prisms and {101} pyramids, that either lack or have only small {110} and {211} facets. According to Pupin (1980), such crystals are mainly found in alkaline to subalkaline granitic rocks and are extremely uncommon in S-type magmatic rocks. The Pupin diagram in Figure 4 illustrates the strikingly bimodal distribution of zircon morphologies in the Toom 1 sample.

Zircons of both groups vary widely in their grainsizes (50–300 μm). Also, the elongation ratios are highly variable, ranging from long, acicular forms to stubby prisms (Figure 3). Most zircons are euhedral, although some appear subhedral owing to rounded edges and tips. The grains of group II are, on average, larger and most are perfectly idiomorphic. In particular the larger group II Zircons carry abundant inclusions of apatite needles as well as inclusions and channels of trapped melt. Group I zircons, in general, have fewer inclusions, but occasionally contain rounded cores that appear to be inherited. Most such core-bearing zircons have high elongation ratios.

In CL images, most zircons feature continuous, oscillatory, magmatic zoning. In some of the larger crystals of group I, there is a significant change of crystal morphology between the core and the rim (e.g. Figure 5, grains a and b). Early growth stages, represented by the inner parts of the crystals, lack the steep {211} pyramids in their zoning patterns, and thus appear to have the group II morphologies of {100} + {101}. The {211} pyramids develop and increase in size during later growth stages, near the crystal rims, commonly overgrowing a resorbed crystal surface (e.g. Figure 5, grain a). It would thus seem
that group II zircons represent an early generation of magmatic zircon, while group I zircons represent a second stage of magmatic zircon growth. Note that Clemens et al. (2011) showed that the Toombullup Ignimbrite magmas underwent multi-stage, polybaric crystallisation with at least two prominent generations of phenocryst minerals; one grown early, in the deep to middle crust, and the other in a very shallow crustal magma chamber, prior to eruption. The two generations of zircon seem to reflect this two-stage crystallisation history.

The CL images also confirm the presence of small (<30 μm), round and irregularly shaped cores in several of the short prismatic crystals (Figure 5, grains d–f). Most of these cores have dark, low-intensity CL signals, implying high U contents, and oscillatory zoning is commonly absent. The zircon domains around the dark cores commonly present either convolute or strong sector zoning, features considered indicative of zircon recrystallisation (Corfu et al. 2003). Only the outer rims of such crystals show typical magmatic zoning and probably represent magmatic overgrowths. Although such core-bearing zircon grains are not rare in the sample, they are small in size and do not constitute a significant proportion of the total zircon volume. From CL images, we estimate that they probably comprise <3% of the total zircon volume.

**Age data**

Areas targeted for U–Pb age dating were carefully selected using the textural information provided by the CL imaging. In total, 43 individual U–Pb age analyses were performed on 17 zircon grains. Of these, 7 analyses were discarded owing to highly irregular time-resolved signals that could not be used for calculation of reliable ages. The irregular signals were most likely due to common Pb-rich inclusions (e.g. apatite; cf. Figure 3) that were not exposed on the surface of the polished zircons and consequently were not detected during CL imaging. The U contents of the analysed spots range from 70 to 849 ppm. However, high U contents (>400 ppm) were only detected in CL-dark cores, and the U contents of the remaining analysed zircons (224 ± 74 ppm) are relatively constant.

The overwhelming majority (~80%, n = 28) of the 36 individual U–Pb age dates obtained for sample Toom 1 are highly concordant and form a well-defined cluster around 380 Ma (Figure 6a). It is noteworthy that all analyses of the texturally distinguished, U-rich, CL-dark cores fall into the group that yielded highly concordant ages. This observation can be best explained by incomplete resorption of inherited zircons and complete resetting of the U–Pb system owing to the high temperatures associated with the melting event that formed the Toombullup magmas (Clemens et al. 2011). The majority of the discordant analyses (n = 5) form an array that trends toward present-day Pb loss. The resulting discordia (Figure 6b; n = 31 with two analyses discarded from calculation because of increased errors) yields an upper-intercept age of 379 ± 9 Ma, a present-day lower-intercept age (within error) and an MSWD of 0.26, which suggests a single, present-day Pb-loss event. Within error, the
upper-intercept age is identical with a concordia age of 377 ± 2 Ma (MSWD = 0.13) defined by the highly concordant age group.

Despite the application of a common Pb correction, three analyses on a single zircon grain (TO11_1, TO11_2 and TO11_4) plot away from the concordia and form a trend toward a compositionally homogenous common Pb source. The presence of this residual common Pb most likely indicates concomitant analysis of domains rich in common Pb or inclusions that were not exposed by polishing and thus not detected by CL imaging.

**CONCLUSIONS AND DISCUSSION**

**Sr and Nd isotope characteristics of the TIC**

Using our new date of 377 ± 2 Ma, we can recalculate the initial Sr isotope ratios and εNd values of the TIC rocks, using the analytical data and present-day isotope ratios published by Clemens *et al.* (2011). Table 2 shows the results. Although the initial 87Sr/86Sr values are virtually identical to those previously obtained, the error bars are now very much smaller, and we can say with certainty that the magmas of the TIC were derived from a compositionally heterogeneous metasedimentary protolith, with clay content in the original sediment being the major variable. These results essentially confirm the Clemens *et al.* (2011) finding that the different units all have near-zero εNd, with initial 87Sr/86Sr in the normal range for S-type granites, confirming the inference that the TIC magmas were derived from metasedimentary protoliths that were constructed from materials not long previously extracted from the mantle, possibly as arc-related volcanic and volcaniclastic rocks. The suggestion is also that the eastern parts of the Selwyn Block, which forms the probable protolith (source material) for the TIC magmas, contains more juvenile volcanic arc material than further west, toward the core of the Melbourne Zone. Also, as mentioned earlier, the metamorphosed, Paleozoic sediments of the region cannot have been deposited on an ocean floor. Rather, they must overlie arc-related rocks in the mainly Proterozoic Selwyn Block. The narrow belts of greenschist-facies Cambrian, mafic volcanic rocks, with associated marine sediments, in this part of Victoria, should not be interpreted as either basement or as representing a primary oceanic layer in the geology. This has significant implications for interpretations of the tectonic development of this part of southeastern Australia.

**Recrystallisation of inherited zircons**

From the CL textures it is evident that there are protolith-derived, inherited cores in the zircons. However, as the analytical data show, these have been totally reset in the U–Pb system, and now have the same ages as the magmatic zircons. It could be argued that the laser beam must have 'drilled' right through these small cores and therefore did not record their true ages. However, the

![Figure 6](image-url)

**Figure 6** (a) Wetherill Concordia diagram showing the U–Pb age data for zircons separated from Toombullup Ignimbrite sample Toom 1; and (b) concordia diagram for 31 discordant zircon analyses from Toom 1. All data were obtained by LA-SF-ICP-MS, and data-point error ellipses are 2σ. See text for further discussion.

**Table 2** Recalculated Sr and Nd isotope data for the analysed rocks of the Tolmie Igneous Complex.

| Unit                        | Sample | 87Sr/86Sr at 377 Ma | ± 2σ calculated | εNd at 377 Ma | ± 2σ assumed |
|-----------------------------|--------|---------------------|-----------------|--------------|--------------|
| Ryans Creek Ignimbrite (low-Ba) | T26   | 0.70904             | 25              | −0.06        | 0.2          |
| Ryans Creek Ignimbrite (high-Ba) | T15   | 0.70985             | 71              | 0.31         | 0.2          |
|                             | T20   | 0.71086             | 11              | 0.44         | 0.2          |
| Toombullup Ignimbrite       | T29   | 0.70804             | 13              | −0.02        | 0.2          |
|                             | T33   | 0.70828             | 12              | 0.19         | 0.2          |

*From data in Clemens *et al.* (2011) and using the date and uncertainty from the present work.
usually high U contents of these cores are clearly recorded in the analyses. We suggest that the cores formed through, or were at least strongly affected by, a dissolution–recrystallisation process during the anatectic stage of magma formation. In particular, U-rich zircons could have fully recrystallised during the anatectic stage at 800–900 °C. Geisler et al. (2007), Rubatto et al. (2008) and Schwartz et al. (2010) showed that, during a high-temperature event, U-rich zircon recrystallises more readily than U-poor zircon, owing to the presence of radioactive damage to its crystal lattice. The presence of convolute zoning together with the scarcity of regular oscillatory zoning in the cores (Figure 5), supports the idea that at least the U-rich domains of the cores (dark in CL) have been recrystallised. Rare U-poor parts within the dark cores, with preserved oscillatory zoning, may have preserved their isotopic compositions (e.g. grain 17 in Figure 5e). However, during analysis, the Pb signals of these domains are weak (owing to low U) and may have been swamped by the signals derived from the adjacent, recrystallised, U-rich zircon.

It seems probable that the inherited zircons were originally larger in size than the present dark, U-rich cores, and also comprised those presumably recrystallised domains in the surrounding zircon that show sector and convolute zoning (see Figure 5, grains d–f). We may be confronted here with an as-yet undescribed dissolution–recrystallisation driven process, during which old zircons largely decomposed and were replaced by newly formed high-U and low-U zircon domains.

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SUPPLEMENTARY PAPERS

Table A1 LA-SF-ICP-MS U–Pb dating methodology CAF. Stellenbosch University.

Table A2 U–Pb age dating results for reference materials Plešovice and M127 obtained during this study by LA-SF-ICP-MS.

Table A3 U–Pb age dating results for sample Tom-1 obtained by LA-SF-ICP-MS.

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