1. Data

The choice of number of calibration standards and validation standards depends on the selected protocol. V2 was the type of the modified protocol.

Calibration standards were prepared at six concentrations levels (25%, 50%, 75%, 100%, 125% and 150%) each day and measured in triplicate.

Validation standards were prepared at four concentration levels (25%, 60%, 100% and 150%) each day and measured in quintet.
Three different series were used for method validation and prepared at the rate of one series per day.

Fig. 1 shows HPLC chromatogram of a validation standard solution containing 0.25 mg ml\(^{-1}\) of AS, 0.325 mg ml\(^{-1}\) of PA, and 0.10 mg ml\(^{-1}\) of GU at optimal condition.

![HPLC Chromatogram](image.png)

**Fig. 1.** HPLC chromatograms of a validation standard solution containing 0.25 mg ml\(^{-1}\) of AS, 0.325 mg ml\(^{-1}\) of PA, and 0.10 mg ml\(^{-1}\) of GU at optimal condition.
2. Experimental design, materials, and methods

AS, PA and GU were supplied by NODCAR. Methanol (HPLC grade), potassium dihydrogen phosphate, and ortho-phosphoric acid 85% were purchased from Merck. A validation matrix solution was prepared by dissolving sodium bicarbonate, citric acid anhydrous, tartaric acid anhydrous, povidone K25, aspartam, and disodium edetate in methanol in order to obtain 24.2, 9.6, 12.3, 1.2, 0.7 and 0.3 mg ml\(^{-1}\), respectively.

The separation was performed on a Phenomenex Luna® CN column, (150 mm \(\times\) 4.6 mm, 5 \(\mu\)m), using an isocratic mode with a mixture of 50 mM potassium dihydrogen phosphate buffer adjusted

| Concentration level | First day | Second day | Third day |
|---------------------|-----------|------------|----------|
| 25%                 | AS(mAU)   | PA(mAU)    | GU(mAU)  |
| 10.689              | 56.742    | 14.456     |          |
| 10.681              | 54.103    | 14.003     |          |
| 10.677              | 54.263    | 14.024     |          |
| 21.156              | 101.945   | 27.198     |          |
| 21.15               | 101.99    | 27.245     |          |
| 21.128              | 101.932   | 27.288     |          |
| 32.535              | 151.523   | 41.696     |          |
| 32.628              | 151.676   | 41.797     |          |
| 32.571              | 151.791   | 41.848     |          |
| 42.494              | 192.624   | 54.309     |          |
| 42.51               | 192.876   | 54.372     |          |
| 42.486              | 192.781   | 54.394     |          |
| 53.399              | 234.045   | 67.952     |          |
| 52.94               | 233.698   | 67.907     |          |
| 52.949              | 234.122   | 67.947     |          |
| 63.259              | 268.373   | 80.922     |          |
| 63.221              | 268.938   | 80.883     |          |
| 63.244              | 268.168   | 80.762     |          |

| Table 1              | Calibration set.                                      |
|---------------------|------------------------------------------------------|
| Concentration level | First day | Second day | Third day |
| 25%                 | AS(mAU)   | PA(mAU)    | GU(mAU)  |
| 10.689              | 56.742    | 14.456     |          |
| 10.681              | 54.103    | 14.003     |          |
| 10.677              | 54.263    | 14.024     |          |
| 21.156              | 101.945   | 27.198     |          |
| 21.15               | 101.99    | 27.245     |          |
| 21.128              | 101.932   | 27.288     |          |
| 32.535              | 151.523   | 41.696     |          |
| 32.628              | 151.676   | 41.797     |          |
| 32.571              | 151.791   | 41.848     |          |
| 42.494              | 192.624   | 54.309     |          |
| 42.51               | 192.876   | 54.372     |          |
| 42.486              | 192.781   | 54.394     |          |
| 53.399              | 234.045   | 67.952     |          |
| 52.94               | 233.698   | 67.907     |          |
| 52.949              | 234.122   | 67.947     |          |
| 63.259              | 268.373   | 80.922     |          |
| 63.221              | 268.938   | 80.883     |          |
| 63.244              | 268.168   | 80.762     |          |

| Table 2              | Validation set.                                      |
|---------------------|------------------------------------------------------|
| Concentration level | First day | Second day | Third day |
| 25%                 | AS(mAU)   | PA(mAU)    | GU(mAU)  |
| 10.807              | 55.015    | 14.218     |          |
| 10.902              | 55.505    | 14.308     |          |
| 10.764              | 54.816    | 14.148     |          |
| 10.714              | 54.726    | 14.164     |          |
| 10.633              | 54.457    | 14.088     |          |
| 25.493              | 121.059   | 32.709     |          |
| 25.473              | 121.107   | 32.727     |          |
| 25.496              | 121.014   | 32.73     |          |
| 25.448              | 121.068   | 32.866     |          |
| 25.547              | 121.517   | 32.878     |          |
| 42.645              | 193.349   | 54.537     |          |
| 42.591              | 193.309   | 54.506     |          |
| 42.582              | 193.325   | 54.503     |          |
| 42.562              | 193.196   | 54.612     |          |
| 42.537              | 193.156   | 54.557     |          |
| 63.282              | 268.914   | 81.076     |          |
| 63.4                | 268.842   | 81.077     |          |
| 63.261              | 269.486   | 80.973     |          |
| 63.31               | 269.579   | 80.962     |          |
| 63.293              | 268.708   | 80.929     |          |

2. Experimental design, materials, and methods

AS, PA and GU were supplied by NODCAR. Methanol (HPLC grade), potassium dihydrogen phosphate, and ortho-phosphoric acid 85% were purchased from Merck. A validation matrix solution was prepared by dissolving sodium bicarbonate, citric acid anhydrous, tartaric acid anhydrous, povidone K25, aspartam, and disodium edetate in methanol in order to obtain 24.2, 9.6, 12.3, 1.2, 0.7 and 0.3 mg ml\(^{-1}\), respectively.

The separation was performed on a Phenomenex Luna® CN column, (150 mm \(\times\) 4.6 mm, 5 \(\mu\)m), using an isocratic mode with a mixture of 50 mM potassium dihydrogen phosphate buffer adjusted
with ortho-phosphoric acid to pH 2.2 as the aqueous component of the mobile phase and methanol in the ratio 85.9:14.1 (v/v) at 1 ml/min flow rate, with UV detector at 275 nm and a column temperature of 45 °C. The injected volume was 20 μL.

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Transparency document

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